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SEDIMENTS

Subject:

Proposed Soil and Sediment Field Sampling Plan for the Otsego City Impoundment
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

Date:
November 22, 2011

Dear Mr. Saric:

Contact:
Michael J. Erickson

The December 2010 Area 2/Otsego City Impoundment Supplemental Remedial Investigation/ Feasibility Study Work Plan (Area 2 SRI/FS Work Plan; ARCADIS 2010a) describes survey activities and soil and sediment sampling designed to: 1) document the current physical conditions of the Kalamazoo River, its side channels, and the river banks in Area 2; and 2) supplement the characterization of the nature and extent of polychlorinated biphenyl (PCB) contamination in sediments, river banks, and floodplain soils in Area 2. The Area 2 SRI/FS Work Plan identifies two investigative phases: survey and reconnaissance in Phase 1 and supplemental soil and sediment sampling in Phase 2. Phase 1 survey work was completed in late 2010/early 2011 and reconnaissance activities – as described in the Field Reconnaissance Plan (Reconnaissance Plan) submitted on April 19, 2011 (ARCADIS 2011) and approved by the United States Environmental Protection Agency (USEPA) on May 2, 2011 – were completed in May 2011.

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This Area 2/Otsego City Impoundment Field Sampling Plan (Area 2 FSP), which was originally submitted on July 29, 2011 and has now been revised to incorporate comments by the agencies, and consistent with the changes requested in the conditional approval provided by USEPA on October 26, 2011. This Area 2 FSP describes the proposed Phase 2 sediment and soil PCB sampling activities. The Phase 2 work is designed to augment historical data and the information gathered during Phase 1 efforts for the purposes of developing an SRI/FS and ultimately supporting a Record of Decision for Area 2. The proposed sediment and soil sampling activities will be completed as soon as possible with agency oversight.

In summary, the proposed investigation activities in Area 2, which lies between the former Plainwell Dam and the Otsego City Dam and includes the Kalamazoo River, its side channels, the floodplain, and the currently and formerly impounded areas, include:

- Floodplain soil sampling within defined soil sampling strata delineated based on information gathered during reconnaissance, with more intensive sampling of low lying areas and former channel areas.
- River bank soil sampling.
- Sediment sampling in the braided channel and off channel areas based on reconnaissance activities.
- Sampling of main channel sediments to provide additional data in areas that are still largely uncharacterized.

The sampling plan components are described in greater detail below, following a summary of the survey and reconnaissance activities used to develop sampling strata.

Summary of Area 2 Survey and Field Reconnaissance (Phase 1)

Between November 15, 2010 and January 12, 2011, ARCADIS completed bank profile surveys and a survey of river transects (including re-survey of a subset of the U.S. Geological Survey [USGS] river transects originally surveyed in 2001 and 2002), and commenced a bank erosion pin study. The bank profile, bank erosion pin, and river channel transect survey locations are shown on figures in Attachment A. Bank profile survey data and a comparison of resurveyed channel transects with earlier USGS data are presented in Attachment B.

The Reconnaissance Plan (ARCADIS 2011) outlined the preliminary desktop study completed by ARCADIS to identify potential strata of interest for soils and areas where additional in-stream sediment information may be needed, and described the methods employed for soil and sediment reconnaissance. ARCADIS used the information acquired during the desktop study to complete soil and sediment reconnaissance in Area 2 between May 9 and May 20, 2011 with Agency oversight. Soil reconnaissance activities included walkover through the area within the SRI/FS boundary (see Figures A-1 through A-4 in Attachment A), collection and description

of soil borings, and observation of vegetation types and boundaries between key features. Sediment reconnaissance included identification of sediment deposits in the braided channels of the impoundment, and collection and description of sediment cores in areas of fine sediment deposition. Field data (including core locations and descriptions) collected during this exercise are presented in Attachment A.

Based on observations made during the reconnaissance, ARCADIS generated a series of maps identifying coverage of different vegetative cover types (Figures 1a through 1d). Also shown on the Figure 1 series are the preliminary boundaries of the geomorphic strata of interest that were identified in the Reconnaissance Plan, locations of reconnaissance soil borings (including locations of those cores where light gray/gray clays or silts were present), and topographic contour intervals at a 0.5-foot contour spacing. A summary of the total area of each of the different vegetation types within the SRI/FS boundary is presented below:

Table 1 - Vegetation Type Summary for the Otsego City Impoundment

Vegetation Type	Area (Acres)
Cattails	73
Cattails/Willow Shrub	3.4
Floodplain Forest	100
Floodplain Forest - ASPEN	2.6
Phragmites	0.25
Purple Loose Strife	11
Reed Canary Grass	14
Upland Forest	62
Wetland Meadow Complex	4.0
Willow Shrub	13
Total Floodplain Area	283

Figure 2 shows locations of sediment deposits identified in the side channels during reconnaissance. Approximately 30 deposits were identified and the approximate extent of each deposit was mapped based on field observations. In addition, Figure 2 indicates whether sediment deposits were found to be fine or coarse and the depth of sediment that was probed in the field.

Summary observations from the survey and reconnaissance work include:

- Soft sediment deposits that occur in the braided area of the impoundment are readily identifiable in the field using the techniques applied during reconnaissance. Maps of depositional areas were developed and indicate that where soft sediment deposits occur, they tend to be relatively small in size and discontinuous along flowing side channels. Channels with low flows and backwater areas tend to have larger areas of soft sediment (see Figure 2).
- A portion of the SRI/FS Study Area¹ on the southern side of the river is a wetland system partially fed by water from channels entering the system from the south of the SRI/FS Study Area. Soil characteristics observed in this area may reflect a natural wetland system that was likely minimally affected by the former impoundment. This observation is supported by existing/historical data, which show a predominance of low PCB concentrations in much of this area. Further details are provided on page 12 of this letter.
- Identified vegetative communities were observed to coincide with some floodplain features visible in historical and recent aerial photographs. Changes in vegetation observed in these areas over time (based on the aerial photographs) appear to reflect the length of time areas have been exposed (i.e., no longer inundated) or the current duration of inundation.
- Because the presence of fine-grained gray materials (i.e., silts and clays) can sometimes indicate the presence of PCB-containing soils, these materials were evaluated in soil borings collected as part of the reconnaissance activities. Table 2 provides a summary of those cores where gray materials were found, including depth interval and soil descriptions. The presence of fine-grained gray materials was found to be spatially variable, sporadic within particular strata, and at varying depths throughout the island areas and in low areas along the river shoreline. Fine-grained gray silt and clay was observed in a number of locations near the surface in fringe wetland areas dominated by purple loosestrife and subject to long-term to permanent inundation. Some gray material, particularly observed in the southern

¹ CH2M HILL defined the Area 2 RI/FS Study Area in its 2003 RI/FS Report (CH2M HILL 2003), and this boundary is incorporated as the SRI/FS Study Area boundary for the purposes of the SRI work. The boundary has been revised in the area of the 12th Street Landfill (Operable Unit 4 [OU4] of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site) to be consistent with the boundary of excavation at OU4.

portion of the impoundment, appears to be native gray clay common in some areas of the river floodplain.

- Bank profiles vary considerably throughout Area 2, ranging from very low lying and gradually sloped banks adjacent to shallow water areas to higher, steeper banks adjacent to deeper water and in areas closer to the dam. Banks will be classified according to the Rosgen Bank Erosion Hazard Index (Rosgen 2006) at a later stage of the SRI. Summary observations of bank profiles are provided below. The bank height observations presented here are relative to the water level at the time of the survey, and are not standardized to a certain flow level. All bank profile transects are shown on the figures in Attachment A.
 - At Transects OCBN-01, 02, 03, 04, 05, 06, and 07 located along the north bank of the main channel moving upstream from the Otsego City Dam, most locations show bank heights of less than 1.5 feet above the surveyed water surface adjacent to shallow water, with bank slopes ranging from near vertical (OCBN-06) to very gradual (OCBN-04).
 - At Transects OCBN-08, 09, and 10 along the north bank of the main channel, bank heights are approximately 1.5 feet above the water surface with gradual bank slopes to water depths of approximately 3.5 to 6 feet. Along the banks on the opposite side of the river on the south side of the main channel (OCBS-08, 09, and 10), bank heights are somewhat higher and steeper, and adjacent water depths are greater.
 - At Transects OCBN-11, 12, 13, 14, and 15 along the north bank of the main channel through the braided section, and OCBS-8, 9, 10, 11, 12, 13, 14, 15 and 16 along the south bank of the main channel, bank heights are 1 to 2 feet higher than the surveyed water surface at most locations, and are relatively steep, with many near vertical bank slopes. Adjacent water depths are typically less than 2 feet.
 - In side channels of the large area to the south of the main channel in the middle of the impoundment (Transects OCBN-18, 19, 20, 21, and 22 along the north banks and Transects OCBS-18, 19, 20, 21, and 22 along the south banks), bank heights tend to be low (1 foot or less above the water surface) with gradual slopes adjacent to shallow water.

- In side channels near the braided upstream section of the impoundment (Transects OCBN-23, 24, 25, 26, 27, 28, 29, 30, 31, and 32 along the north banks and Transects OCBS-23, 24, 25, 26, 27, 28, 29, 30, 31, and 32 along the south banks), bank profiles are variable depending on location. Typical bank heights are between 1 and 2 feet above water surface. Some locations have steep banks, but many locations have low banks with gradual slopes.
- When compared to the 2001-2002 USGS transect survey results, the recent channel survey results indicate a range of changes have occurred in the approximately 10-year interval between surveys. Minimal changes were evident in some areas, suggesting that the survey data are comparable among events, while substantial differences in bed elevation and configuration were evident in other areas, suggesting either channel migration or areas of transient erosion and deposition.
 - USGS Transects G3, G5, G7, and G9 (resurvey transects OCRT-02, 04, 07, and 10, respectively; shown in plan on the figures in Attachment A and in profile in the figures in Attachment B) located between the Otsego City Dam and the current extent of the dam backwater² show that the channel cross-section has maintained a consistent shape and position with variation in bed elevation ranging from near zero (little change) to up to 2 feet of either bed loss or accumulation. Most surveyed points along these transects compare within 1 foot elevation of the USGS survey.
 - USGS Transects C9, F5, F12, and B2 (resurvey transects OCRT-17, 31, 36, and 38, respectively) in the braided section indicate the channels in this area continue to evolve. Comparison of the ARCADIS and USGS transects suggest channel cross-section shape and position has changed substantially over the past 10 years, with some locations indicating lateral movement of the channel in addition to significant variations in bed loss and accretion, depending on location.
 - USGS Transects A2 and A1 (resurvey transects OCRT-42 and 43, respectively) located just downstream of the former Plainwell Dam spillway show a significant change in sediment bed elevation. At Transect A2, results

² USGS 2004 indicates that the current impoundment extends back as far as USGS transect G10, just over 1,100 feet upstream of G9 and as indicated in Figure 1

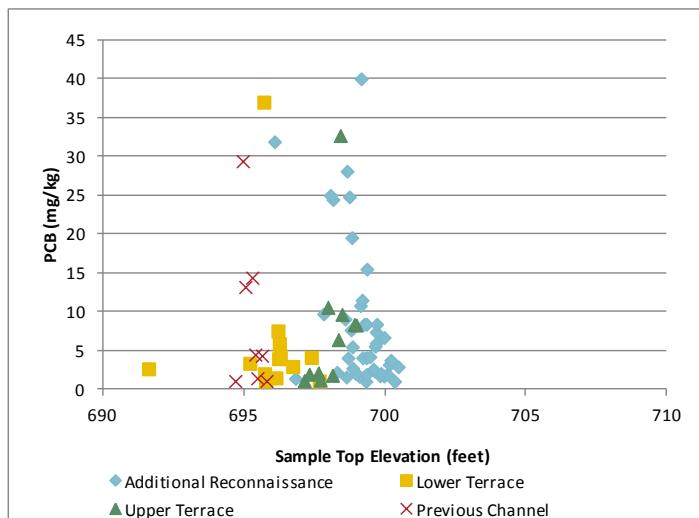
indicate a loss of 1 to 2 feet of sediment across most of the transect. At Transect A1, up to 7 feet of sediment appears to have accumulated on the east side of the channel downstream of the spillway, with approximately 3 feet of bed loss on the west side. The large change in bed elevation in this area may have partially resulted from sediment transport during the October 2008 flood.

The reconnaissance information, when coupled with existing data, provide a basis for a stratified PCB sampling design based on geomorphic and vegetative strata. Variations in geomorphic characteristics and type of vegetation reflect differences in depositional history. To guide identification of strata for sampling design, existing PCB concentrations were evaluated within the geomorphic features identified in the Reconnaissance Plan and within areas of similar vegetation cover observed during reconnaissance.

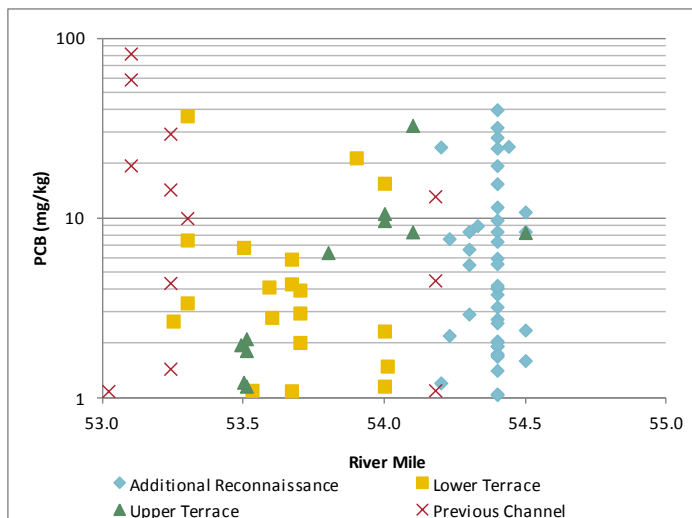
Varying sample density per acre, and the high proportion of non-detections in the overall dataset obscure to some extent the differences in PCB data populations among strata (for example, 49% of the samples had non-detected PCB, and 86% of the samples were either non-detect for PCBs or had PCB concentrations less than 1.0 mg/kg). Overall, the four geomorphic strata identified in the Reconnaissance Plan (i.e. Previous Channels/Previous Side Channels, Lower Terraces, Medium/Upper Terraces, and Additional Reconnaissance Areas as identified on Figure 6 of the Reconnaissance Plan) seem to represent two populations of PCB concentration. The Lower Terrace and Additional Reconnaissance area PCB concentrations are statistically similar to one another and both are statistically higher than the PCB distributions in the Previous Channels and Upper Terraces areas. Considering only samples with PCB greater than 1.0 mg/kg (to more clearly identify strata where elevated PCB concentrations would be expected to occur), the geomorphic strata show some separation. For the Upper Terraces, which historically have been the most densely sampled in certain areas as a result of USEPA's prior radial sampling, the majority of samples with PCB greater than 1.0 mg/kg were located around river mile 54.4, and at estimated sample elevations (surface elevation minus sample top depth) between 698 and 700 feet above National Geodetic Vertical Datum of 1929 (NGVD29) (see graphs below; note that USEPA sediment samples are not shown on the figure by elevation). Lower terrace samples with PCB greater than 1.0 mg/kg occur near and downstream of river mile 54, and occur primarily at sample elevations less than 698 feet NGVD29. Within areas designated as Upper Terraces, the higher concentrations occur at higher elevations of 698 to 699 feet NGVD29. Additional

graphs showing all PCB data (in addition to those greater than 1 mg/kg) are also presented.

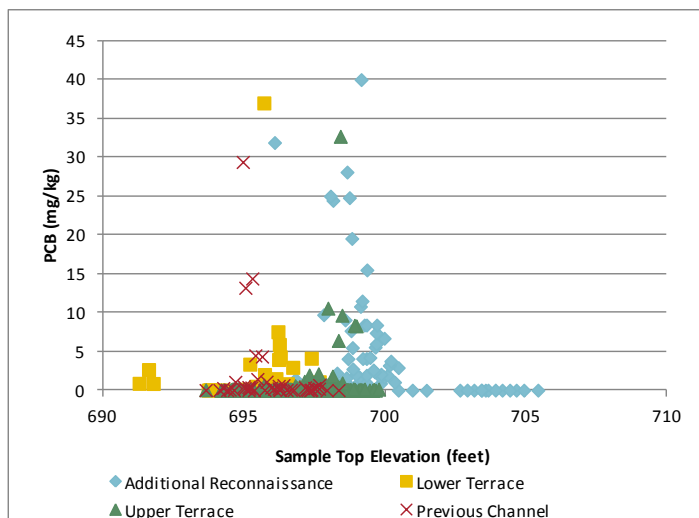
All PCB Greater than 1.0 mg/kg



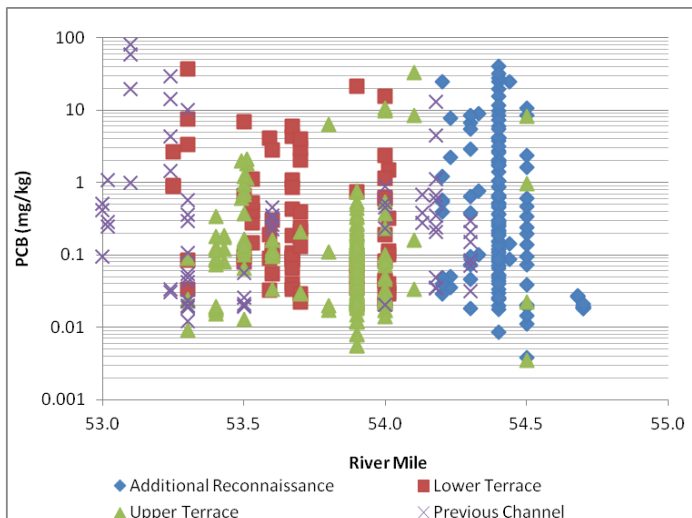
All PCB Greater than 1.0 mg/kg



All PCB



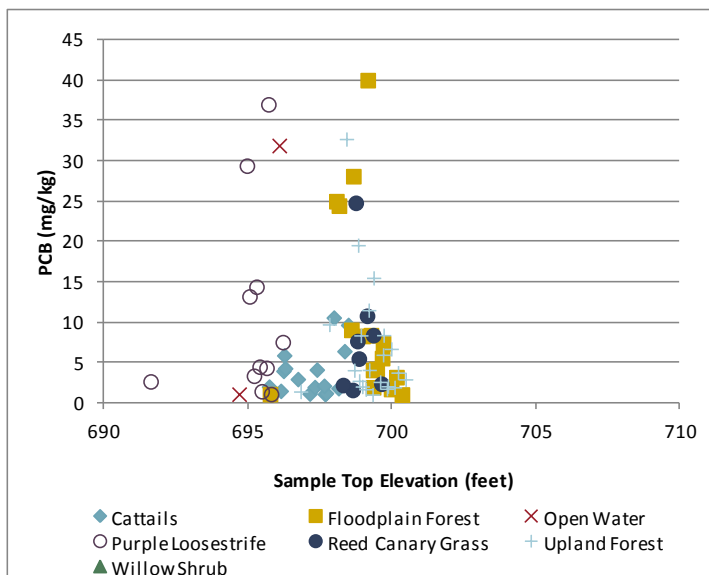
All PCB



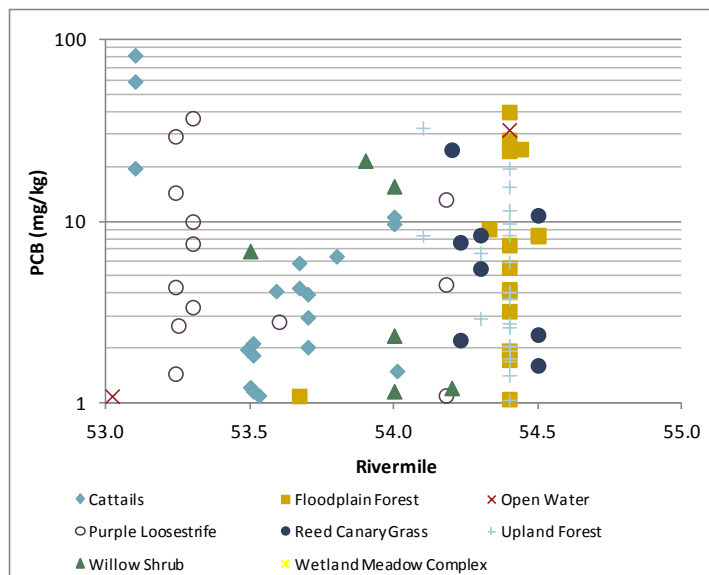
Note: The sample that approaches 100 mg/kg is a USEPA sediment sample located on the north side of the river upstream of the dam. Elevation data are not available for the USEPA sediment samples so these samples are not shown on the elevation graph.

When stratified by current vegetation type at the sampling location, differences in PCB concentrations greater than 1.0 mg/kg suggest a possible basis for stratification based on vegetation type (see graphs below). For example, PCB concentrations greater than 1.0 mg/kg in the area of river mile 54.4 comprised samples from the floodplain forest, upland forest, and reed canary grass; three vegetation types associated with the more upland, less inundated environments of Area 2. This area may have been historically affected primarily by over-bank flooding rather than by the impoundment. Downstream of river mile 54.4, PCB concentrations greater than 1.0 mg/kg occur in soil vegetated with cattails and purple loosestrife, flora typically occurring in lower, more saturated areas, if not in standing water. This is corroborated by the sample elevation data, which show that all the samples below 698 feet NGVD29 with PCB concentrations greater than 1.0 mg/kg (except two upland forest samples from depth increments of 36- to 45-inches and 24- to 36-inches) were collected from areas of cattail, purple loosestrife, or open water.

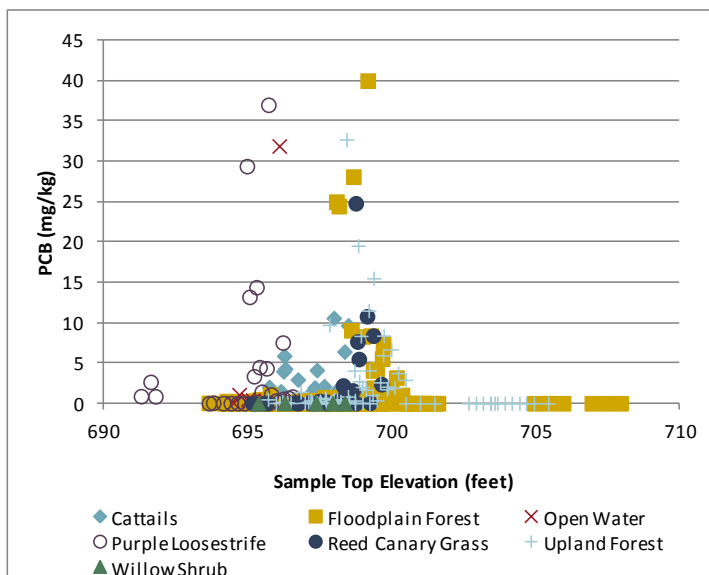
All PCB Greater than 1.0 mg/kg



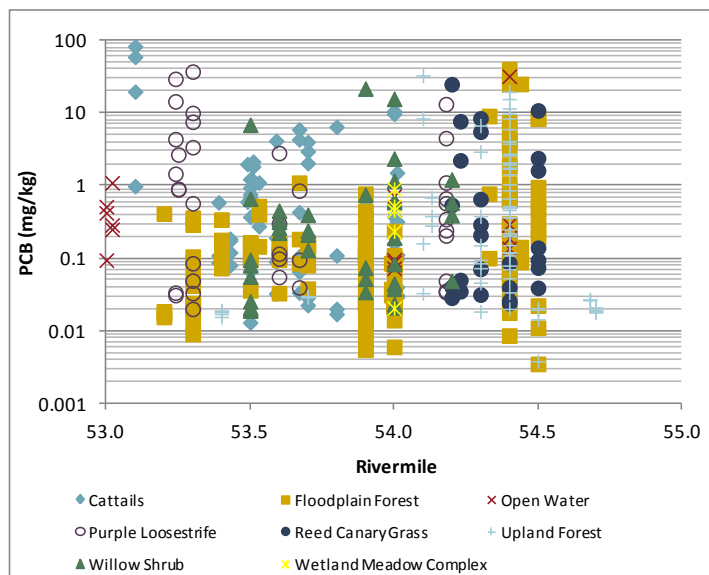
All PCB Greater than 1.0 mg/kg



All PCB



All PCB



Note: The sample that approaches 100 mg/kg is a USEPA sediment sample located on the north side of the river upstream of the dam. Elevation data are not available for the USEPA sediment samples so these samples are not shown on the elevation graph.

Refinement of Soil Sampling Strata

A goal of the reconnaissance effort was to collect data needed to refine potential soil sampling strata for sampling design. To develop the targeted Phase 2 sampling locations, ARCADIS reviewed the spatial correspondence of reconnaissance information shown in Figure 1 (vegetation types, soil boring information, and topographic contours) and the original geomorphic feature boundaries created from historical aerial photographs during the desktop review. The original boundaries were preserved unless sufficient information was available to justify relocation. If a boundary was relocated, it was either moved to correspond to changes in vegetation types, or parsed into smaller areas based on vegetation type. In a limited number of instances, adjacent strata were collapsed into a single stratum for sampling design purposes if reconnaissance information or review of existing PCB data did not support treating them separately. For example, the distinction between former channel and former main channel strata was removed and these were treated as a single stratum. Where strata were parsed based on vegetation type, this was predominantly due to locations of emergent wetland vegetation boundaries.

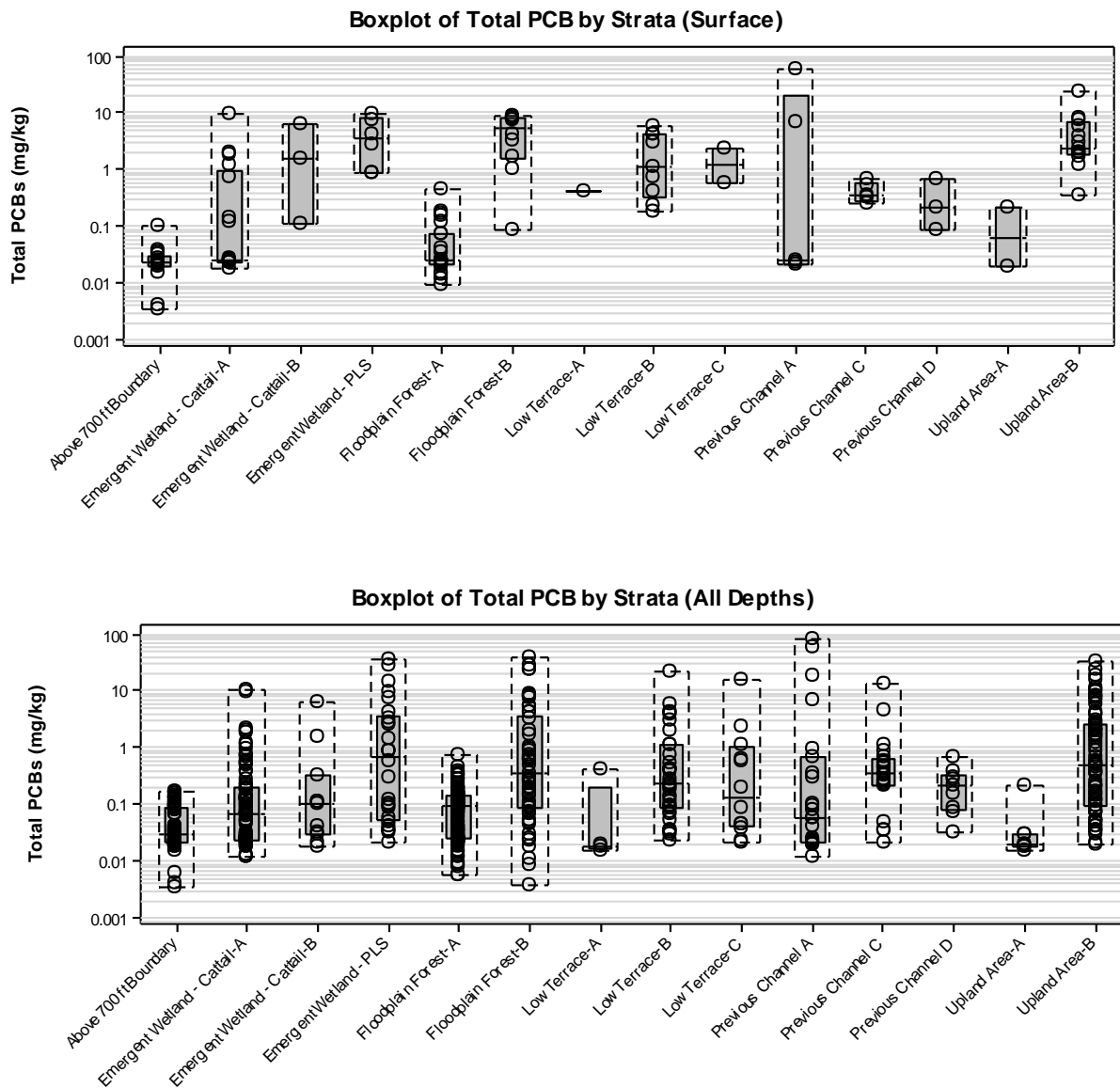
The following types of strata were identified:

- Upland Area Areas with well-established upland forest vegetation that have not shown inundation in the past
- Floodplain Forest Areas with floodplain forest vegetation that fall within the 100-year floodplain, but do not appear to have been inundated in the past
- Low Terrace Areas that have shown some inundation in the past
- Emergent Wetland Areas of current emergent wetland-type vegetation; two types of emergent wetland were identified – purple loosestrife areas, which tend to be in areas of permanent inundation, and cattail areas, which did not appear to be permanently inundated
- Previous Channel Areas that appear to have been channels in previous years

Strata are identified on a unit by unit basis in Figures 3a through 3d. Several of the strata are comprised of several separate (non-contiguous) groups of units which exhibit similar characteristics. Based on an evaluation of sample density and historical PCB distribution within these areas, it is apparent that substrata can be created based on geographical grouping of like areas within the strata. Table 3 indicates the groupings that were developed for each of the sub-strata:

A summary of existing PCB data within each of these sub-strata is shown in the box plot in Figure 4, below. These data show some differences within a given stratum based on geographical location within Area 2. For example, PCB concentrations in floodplain forest soil are lower in the downstream portion of Area 1 (vegetation group A) and higher in the upstream end (Floodplain Forest B). This indicates that the stratification process resulted in a meaningful differentiation in most cases.

Figure 4: Boxplots of Existing Total PCB Concentration by Sub-Strata



To the south of the river, there is a large forested floodplain area (identified as Floodplain Forest areas 1, 2, and 3 in Figure 3c) where the majority of the soil cores collected and observed during reconnaissance contained organic peats and sands. In addition, groundwater elevations observed in boreholes suggest a general groundwater flow towards the river. The high water table and the northward flow in drainage channels south of the SRI/FS Study Area appear to be important hydrological inputs to this wetland system.

Existing PCB sample data in this forested floodplain area (sample locations and concentrations are illustrated on Figure 3c) suggest that this area has not been subject to prolonged periods of inundation or sediment deposition from the Kalamazoo River. There are 28 prior sampling locations in this area – the maximum reported total PCB concentration is 0.77 mg/kg, with a core maximum mean of 0.20 mg/kg and a standard deviation of 0.19 mg/kg (and mean and standard deviation for all PCB data of 0.10 mg/kg and 0.11 mg/kg, respectively). Using the maximum PCB concentration value for each location in the USEPA software program ProUCL, the estimated 95% upper confidence limit (UCL) for the mean is 0.27 mg/kg (using the recommended approximate gamma 95% distribution). Using all PCB concentration values, the estimated 95% UCL for the mean is 0.14 mg/kg (using nonparametric Chebyshev 95%). Assuming the 28 samples are representative and are drawn from a normal population, 99.9% of the time an individual sample from this area should have a PCB concentration less than 0.81 mg/kg. As a result of the apparent low degree of PCB impacts to soils in this wetland system, no Phase 2 sampling is proposed in this area.

Based on the existing PCB concentration distribution in the floodplain soil sampling strata, the Phase 2 sampling locations will target those areas and strata where there are either few existing samples or higher observed PCB concentrations. Areas where sufficient data coverage already exists or where PCB concentrations are low will be excluded. Based on this approach, no sampling is proposed in the areas above 700 feet NGVD29 (which includes the upland area to the east and downstream of the former Plainwell dam) and in the Floodplain Forest areas 1, 2, and 3.

Other Compounds

Data for constituents other than PCBs are currently available in Area 2. Available sediment and soil non-PCB data for Area 2 are presented in Attachment C. These include Target Compound List and Target Analyte List (TCL/TAL) results from one soil core (KF3-1) collected by BBL in 1993 and dioxin/furan data for four soil and nine

sediment samples collected by USEPA contractors in 2001. The locations of these sample points are identified in the databox figure presented in the Area 2 SRI/FS Work Plan.

Available dioxin/furan data for fish, including data from samples collected by the State in 2009, will be incorporated into the Area 2 SRI Report.

To supplement the existing non-PCB constituent data, samples from a subset of the Phase 2 soil and sediment cores will be collected and analyzed for TCL/TAL and dioxin/furans in addition to PCBs. Details on the proposed non-PCB sampling in Area 2, which was developed in consultation with USEPA and MDEQ, are presented in this sampling plan.

Description of Supplemental Data Needs and Sampling Objectives

Section 4.1 of the Area 2 SRI/FS Work Plan lists the following data needs for soils in Area 2:

- Soil PCB concentrations representative of specific geographic areas and geomorphic features of Area 2.
- PCB concentrations in floodplain soils representative of elevation strata and/or historical depositional strata within the formerly impounded area and low-lying areas of the floodplain in the braided section where existing spatial coverage is low.
- PCB concentrations in floodplain soils in areas proximate to the bank of the main channel upstream of the formerly impounded area where floodplain deposition may have occurred in low lying areas proximate to the main channel during the period of increased braiding following the former Plainwell Dam drawdown, and where spatial coverage is low.
- PCB concentrations from bank soil samples representative of bank types along the main channel and in the braided channels.
- Bank types and stability conditions throughout Area 2. (This is being addressed through use of the survey data presented in Attachment B, and the ongoing erosion pin study, per the Area 2 SRI/FS Work Plan.)

The following data needs are listed for sediments in the Area 2 SRI/FS Work Plan:

- Current surface sediment PCB concentrations throughout Area 2.
- Sediment PCB concentrations representative of specific geographic areas (channel braids, impounded area, back water areas) and geomorphic features (near-shore sediment deposits, point bars, etc.) of Area 2.

Based on reconnaissance observations, review of the survey data presented above, and locations of existing soil and sediment PCB samples, the following additional data need for sediment has been identified:

- Sediment PCB data from the area just downstream of the former Plainwell Dam spillway to assess PCB concentrations in the sediment accumulated in this area since the USGS survey.

Based on these needs, and USEPA's request to include non-PCB analyses for a subset of samples, the following sampling objectives are defined:

- Collect floodplain soil samples for PCB analysis to obtain data representative of specific strata defined based on the desktop study of historical inundation and former channel areas, and as refined by field reconnaissance information, especially where sampling strata are under-represented by existing sample points, or current spatial coverage is inadequate.
- Collect soil samples for PCB analysis along the banks of the main channel and side channel areas with erosional bank conditions to obtain data representative of bank soils to support estimations of PCB loading from the banks to the river (bank profile locations with low gradually sloped banks adjacent to shallow water will not be sampled).
- Collect sediment samples from main channel sediments in areas selected to improve spatial coverage when combined with existing samples.
- Collect sediment core samples for PCB analysis from within selected soft sediment deposits in the side channels identified during reconnaissance (it is assumed that the majority of these samples will be classified as fine sediments).

- Collect sediment core samples from the east side of the main channel just downstream of the former Plainwell Impoundment spillway for PCB analysis.
- Analyze a subset of the collected soil and sediment samples for TCL/TAL analytes and dioxin/furans.

Proposed Phase 2 Sampling Approach

The proposed sampling plan incorporates the following approaches by medium and area to fulfill the sampling objectives:

- Sampling for floodplain soil within the vegetative strata defined in Phase 1
- Systematic sampling for bank soil across areas with eroding banks
- Random sampling for main channel sediments in the limits of the current dam backwater
- Biased sampling of the side-channel soft sediment deposits identified during reconnaissance

The components of the proposed investigation are described in detail below and are summarized on Table 4 and Figure 5. The spatial extent of supplemental sampling is the SRI/FS Study Area, excluding the natural wetland system and floodplain forest area in the southern portion of the SRI/FS Study Area (Floodplain Forest Areas 1, 2, and 3), and areas above 700 feet NGVD 29 (including the upland forest located to the east of the former Plainwell Dam) as shown in Figure 1-a.

Sample Locations

Floodplain Soil Samples

Floodplain sample locations were selected with a goal of collecting sufficient numbers of samples to represent the individual stratum. Based on discussions with USEPA and MDEQ, proposed sample density per acre is greater in areas that are more likely to contain PCBs, specifically the Previous Channel, Low Terrace, and Purple-Loosestrife sampling strata. These areas will be characterized with a sample density of approximately 1.5 samples per acre. Additionally, per agency request, Floodplain Forest 7a will also be sampled at a density of 1.5 samples per acre. The

other proposed sampling strata will be sampled with a 1 sample per 2 acre density. Sampling locations were developed by overlaying a 100-foot sampling grid across the SRI/FS Study Area and then randomly selecting the appropriate number of grid nodes to achieve the required number of samples per strata to achieve the target sampling density. Final locations were constrained so that proposed sample locations were a minimum of 100 feet from existing sample locations. No supplemental sampling is proposed for two of the strata, the Areas above 700 feet in elevation, and the sub-stratum Floodplain Forest A, where previous PCB sample locations are of sufficient density and concentrations are consistently low (i.e., no samples greater than 1.0 mg/kg).

The MDEQ, based on their reconnaissance, has described locations at which floodplain soils were observed to overlay layers of fine-grained gray-colored soil at depth that may indicate PCB-containing materials. Based on discussions with MDEQ, examples of these areas include "Previous Channel 4" and "Previous Channel 5" shown in Figure 3-b³. Reconnaissance observations were discussed with MDEQ to select a subset of the proposed soil sampling locations where additional reconnaissance and potentially analysis of these deeper materials will be conducted. A total of 20 locations (approximately 15 percent of floodplain sample locations) will be subject to deeper coring (Table 5). At these locations, core samples will be extended to at least 60 inches below ground surface. Samples from the top 48 inches will be submitted for laboratory analysis. If fine grained gray clay materials are observed at depths below 48 inches, samples from additional depth intervals may be submitted for PCB analysis, and if so, samples will be continuous through the core down to the bottom-most depth interval of any deeper layers selected for analysis. The selection of any sample depth intervals below 48 inches for PCB analysis will be made in the field based on a judgment of whether the materials appear likely to potentially have elevated PCB concentrations based on observation of soil type. If depth intervals selected for analysis extend to 60 inches, additional efforts will be made to extend cores to deeper depths to delineate the extent of the layer of interest. The depth of sample will be subject to practical limitations of the sampling equipment and lexan core tubes available in the field. In these locations, the sample depth may be extended by hand augering through sample depths recovered with an initial core, and then extending the sample with an additional deeper core sample. In these

³ Personal conversation between Mike Erickson, ARCADIS, and John Bradley, MDEQ. Oct. 11, 2011.

cases, the resulting sample will be logged as a continuous sample, collected in two separate core sections.

The final proposed sample locations are shown on Figure 5 and summarized in Table 4. This proposed plan will result in 134 floodplain soil sample locations. An estimated 563 samples will be analyzed for PCBs (assuming an average core depth of 32 inches based on previous data) and an estimated 134 samples will be analyzed for total organic carbon (TOC) and particle size distribution. Table 3 provides a summary of existing and proposed floodplain sample densities on a stratum and unit-by-unit basis.

As discussed above, a subset of the sediment and floodplain soil core samples will be taken and analyzed for TCL/TAL and dioxin/furan compounds. These samples are discussed below.

Bank Soil Samples

Bank soil samples will be collected from the top-of-bank to refusal at the locations shown on Figure 5 to characterize the nature and extent of PCBs in bank soils and estimate PCB loadings from bank erosion. Most sampling locations coincide with bank erosion pin locations that are already in place following Phase 1 fieldwork activities. Proposed bank soil core locations are primarily located along the main channel of the Kalamazoo River with less spatially intensive sampling in the side channels. In some areas, bank slopes are gradual with no defined top of bank location evident. In these areas, an appropriate location for sampling within a distance of 30 feet from the edge of water will be selected in the field in collaboration with Agency oversight personnel.

Bank characterization will be conducted at 34 stations on each bank for a total of 68 individual bank sample locations.

Bank sampling will generate an estimated 286 samples (based on an average core depth of 32 inches), which will be analyzed for PCB Aroclors and 68 samples analyzed for TOC and particle size distribution.

Sediment Samples

Main channel sediment samples upstream of the dam to the approximate extent of the current dam backwater (see note marking the approximate location on Figure 1)

were randomly distributed within segments with no existing samples. MDEQ requested that a subset of the main channel sediment locations be moved so that they are located next to proposed bank sample locations within 5 feet of the toe of bank. Eight main channel sediment sample locations were repositioned in this manner and their locations are identified in Figure 5.

Upstream of the current dam backwater, the main channel is swifter flowing with and contains predominantly coarse-grained materials, therefore locations of main channel sediment samples were targeted toward channel margin and point bar features likely to contain finer grained sediments.

Three sediment core locations are proposed along the east side of the pool downstream of the former Plainwell Dam spillway where substantial sediment accumulation since 2001-2002 is evident (see summary of reconnaissance observations discussed above and on Figure 5). If the planned locations do not yield any core recovery, the sample locations will be repositioned in the field up to a distance of 50 feet.

Side channel sediment cores will be collected at locations in areas of sediment deposits identified by ARCADIS personnel during reconnaissance, and supplemented by additional locations requested by MDEQ based on incorporation of their additional reconnaissance information. As summarized previously in the presentation of reconnaissance information, a number of fine grained sediment deposits were identified in the side channel areas and are presented on Figure 2. Core samples were preliminarily proposed in 12 of the deposits identified during reconnaissance as well as areas of the numerous braided channels where no existing sample locations existed. Subsequently, MDEQ proposed several modifications to the preliminary plan and in response, additional a number of samples were added in other fine-grained sediment areas and several of the preliminarily proposed locations were adjusted. In all, an estimated 55 sediment cores will be collected from the side channels at locations as shown in Figure 5. Figure 6 indicates how the sediment samples are located relative to the features identified during Phase 1 survey and reconnaissance activities.

In the current configuration, of the 55 side channel samples, 47 (85%) sample locations are positioned in fine materials (including 20 which are located in fine material deposits identified during reconnaissance activities – see Figure 2) and 8 (15%) samples locations are positioned in coarse materials.

It is anticipated that data from off-channel areas disconnected from flowing areas and/or otherwise relatively isolated would not be used in the development of surface-weighted average concentration calculations (SWACs) for Area 2. Specifically, data from the small ponded area to the north of the Gun River and the isolated channel area on the very eastern side of Area 2 (downstream and east of the former Plainwell Dam) will only be used to define the features and not in the calculation of the SWAC.

A total of 81 sediment sampling locations are proposed, which will yield an estimated 511 samples for PCB analysis and 81 for TOC and particle size distribution.

A subset of sediment core samples will be taken and analyzed for TCL/TAL and dioxin/furan compounds as described below.

In addition, sediment re-sampling activities identified in Section 4.2.2 of the Area 2 SRI/FS Work Plan will be performed concurrently with the above sediment sampling activities. This sampling is described below:

- Surface sediment samples (0- to 2-inch increment) are proposed to be collected from the approximately 22 sample locations originally sampled in 1993 and resampled in 2000 (Figure 4-1 in the Area 2 SRI/FS Work Plan). The goal of this sampling is to ascertain whether or not changes in surface sediment PCB concentrations have occurred since 1993 given the passage of time and the construction of the removal action at the former Plainwell Impoundment. Further sediment sampling activities to collect full cores from other locations for analysis, including deeper intervals, is anticipated and will be performed during Phase 2 activities, described below. The re-sampling locations are specific to just those locations sampled twice previously.
- At each of the targeted re-sample locations, a core will be collected by driving Lexan® tubing into the sediment until refusal, creating a vacuum, and retrieving the sediment. Each core collected will be photographed and described using the Uniform Soil Classification System (USCS). The upper 2 inches of sediment will be sectioned from the core and submitted for laboratory analysis of PCB Aroclors; TOC analysis and particle size distribution.

Sampling and Analysis Methods

All sampling will be performed per the methods described in the Multi-Area Field Sampling Plan (ARCADIS BBL 2007), and consistent with the Area 2 SRI/FS Work Plan (ARCADIS 2010a).

All soil samples will be submitted to the laboratory for PCB Aroclor analysis. The surface sample from each core will also be analyzed for TOC and particle size distribution.

All sediment samples will be submitted for laboratory analysis of PCB Aroclors; surface samples will also be submitted for TOC analysis and particle size distribution. A subset of approximately 20% of the sediment cores to be collected in this task will be analyzed for particle size distribution and TOC over the entire core (i.e., all depth increments).

PCB analysis will be conducted using the TestAmerica protocol for USEPA Method 8082 approved by USEPA in the Multi-Area Quality Assurance Project Plan (ARCADIS 2010b).

A subset of sediment, bank, and floodplain soil core locations (as identified in Table 5) will additionally be sampled for TCL/TAL constituents and dioxin/furan as follows:

- For all core locations, the recovered core will be segmented and homogenized, and the samples split. The PCB, TOC, and particle size distribution samples will be collected and submitted from the appropriate intervals.
- At 20% of core locations, additional material will be collected, as necessary (to be determined in the field based on core stratigraphy) for potential future analysis of Volatile Organic Compounds (VOCs), Semi-volatile Organic Compounds (SVOCs), pesticides, metals, mercury, and dioxin/furan. Additional coring may be necessary to provide enough volume for sample intervals of 6 inches or less, for example the upper foot of material, or thin distinct layers at depth.
- For half of those locations (10% of total core locations), samples will be collected from the remaining material from the uppermost two depth intervals and submitted to the laboratory for VOC and mercury analyses, to accommodate the relatively short holding times for these analyses (14 days and 28 days, respectively).

- The remaining material from all cores and segments will be placed in jars, labeled, and retained temporarily in frozen storage pending results of the PCB analyses.
- Subsequent samples will be retrieved from frozen storage and submitted to the laboratory for SVOC, pesticide, metals, and dioxin/furan based on the results of the PCB analyses of all cores. Thirty samples will be analyzed for SVOC, pesticide, metals, and dioxin/furan to represent samples with a range of PCB concentrations, and the remaining archived samples will be disposed of. Samples will be submitted to represent three intervals of PCB concentrations. The specific concentration intervals will be selected and confirmed after review of the PCB results; however, the following are preliminarily proposed:
 - Non-detect to 0.50 mg/kg 10 samples
 - 0.5 mg/kg to 5.0 mg/kg 10 samples
 - Greater than 5.0 mg/kg 10 samples
- All sample handling and analysis will be performed in accordance with the Site-Wide FSP and QAPP.

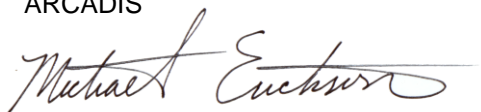
Dioxin/furan analysis is not addressed in the Multi-Area Quality Assurance Project Plan (ARCADIS 2010b), therefore, the dioxin/furan analysis will be conducted using the TestAmerica protocol for *“Analysis of Polychlorinated Dioxins/Furans by High Resolution Gas Chromatography/High Resolution Mass Spectrometry Based on methods 8290, 8290A, 1613B, 23, 0023A, and TO-9A”*. The Standard Operating procedure for this analysis is provided in Attachment C.

Schedule

I will contact you to discuss this proposed sampling plan. ARCADIS has initiated sampling in collaboration with agency oversight personnel and will complete the field work as soon as possible once a USEPA-approved plan is agreed upon. Sampling will be completed expeditiously, subject to scheduling field crews, confirming availability of Agency oversight personnel, and obtaining any necessary additional access agreements. Our goal is to complete all field work by the end of October 2011 and to receive laboratory results from submitted samples in November and December. Following receipt of initial results, archived samples for additional non-

PCB analysis will be submitted to the laboratory. Once all results are received, a meeting will be scheduled with USEPA and MDEQ to discuss the results and potentially additional sampling needs.

Sincerely,
ARCADIS



Michael J. Erickson, P.E.
Vice President

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Enclosures:

Table 1	Vegetation Type Summary for the Otsego City Impoundment (in text)
Table 2	Sample Locations with Gray Silt or Clay
Table 3	Summary of Existing and Proposed Sample Densities by Strata
Table 4	Summary of Proposed Sediment and Soil Sampling Plan
Table 5	Non-PCB and Deeper Sample Analysis

Figures 1a-1d	Identification of Floodplain Areas by Vegetation Type
Figure 2	Identified Sediment Deposit Areas
Figures 3a-3d	Floodplain Sampling Strata
Figure 4	Boxplots of Existing Total PCB Concentrations by Sub-Strata (in text)
Figure 5	Proposed Sample Locations – Area 2
Figure 6	Proposed Sediment Sample Points

Attachment A	Data from Phase 1 Reconnaissance
Attachment B	Bank Profile Survey and USGS Transect Re-Survey Profiles
Attachment C	Area 2 Non-PCB Sampling Information

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Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 2 - Sample Locations with Gray Silt or Clay

Vegetation Type	Sample ID ¹	Core Interval (Inches)	Core Description
Open water	OCRT-03 (2+98)	5 - 16	Gray silty clay, trace organics (root fibers), moderate odor
	OCRT-14 (0+06)	0 - 10	Gray silty clay, trace organics (roots), slight odor
	OCRT-29 (0+85)	14 - 26	Gray silty clay, trace fine sand, no odor
	OCRT-31 (0+28)	20 - 24	Gray clayey silt, slight odor
	OCRT-31 (0+52)	30 - 39	Gray silty clay, trace organics (wood/twigs), slight odor
	OCRT-37 (0+96)	4 - 16	Gray grading to gray brown clayey silt, trace organics (roots and shell fragments), slight odor
	OCRT-37 (1+09)	18 - 22	Gray silty clay, trace organics (twigs and roots)
	OCRPS-4	29 - 30	Gray silt, little fine sand
	OCRPS-8	6 - 23	Gray silty clay, odor
	OCRPS-16	6 - 14	Gray clay silt, trace fine sand, sheen, slight odor
	OCRPS-17	24 - 26	Gray silty clay, sheens, odor
	OCRPS-21	5 - 9	Light gray silty clay, slight odor, trace sheens
	OCRPS-23	6 - 16	Gray silty clay, trace fine sand, odor, sheens
	OCRPS-33	24 - 30	Gray silty clay, trace shells, no odor, no sheen
	OCRPS-43	25 - 37	Light gray to gray silty clay
	OCRPS-52	16 - 21	Gray coarse silty clay, trace fine sand, trace organics
	OCRPS-65	24 - 40	Gray clayey silt, trace fine sand
Cattails	OCRPF-08	7 - 36	Gray clay (Dark yellowish brown)
	OCRPF-45	0 - 36	Gray sandy silt (Very dark grayish brown)
	OCRPF-59	0 - 36	Gray silt and clay, trace sand, some organics/detritus
	OCRPF-107	4 - 10	Gray silty clay
	OCRPF-110	12 - 18	Gray clay
	OCRPF-111	7 - 36	Black to gray silt with organics, changing to brown with depth (Very dark grayish brown)
	OCRPF-112	4 - 20	Gray clayey silt with organics
		30 - 36	Gray sandy silt
	OCRPF-117	0 - 30	Gray silty clay, trace sand
	OCRPF-121	0 - 4	Light brown and gray clay with organic detritus
		4 - 24	Light brown and gray clay
		24 - 36	Black detritus and light gray clay
	OCRPF-128	0 - 4	Peat, gray silt with organics
	OCRPF-129	3 - 9	Gray silt with organics
	OCRPF-145	33 - 36	Clay, becoming more gray
	OCRPF-147	20 - 30	Gray and black clay with detritus
		30 - 36	Gray clay with detritus
Floodplain forest	OCRPF-80	4 - 28	Gray silt with organics, trace sand
	OCRPF-81	24 - 36	Gray sandy silt
	OCRPF-82	30 - 36	Gray silty clay
	OCRPF-92	14 - 36	Gray sandy silt
	OCRPF-94	24 - 36	Gray sandy silt
	OCRPF-105	0 - 30	Grayish clayey silt with organic detritus
	OCRPF-123	1 - 16	Gray clay (Dark yellowish brown)
	OCRPF-130	0 - 3	Organic silt and detritus, gray clayey silt (Black)
	OCRPF-148	30 - 32	Gray and black organic clay
		32 - 36	Gray silty clay

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Table 2 - Sample Locations with Gray Silt or Clay

Vegetation Type	Sample ID¹	Core Interval (Inches)	Core Description
Purple loosestrife	OCRPF-52	11 - 24	Light gray sandy silt, trace sand
	OCRPF-118	0 - 4	Gray sandy silt
	OCRPF-149	0 - 36	Light gray silt
	OCRPF-152	1 - 36	Light gray silty clay
Reed canary grass	OCRPF-48	30 - 36	Gray sandy silt (Very dark gray)
	OCRPF-57	11 - 36	Gray silty clay, some sand
	OCRPF-58	32 - 36	Gray sandy silt
	OCRPF-73	0 - 30	Grayish silty clay (Very dark grayish brown)
		31 - 36	Grayish silty clay (Very dark grayish brown)
Upland forest	OCRPF-50	30 - 36	Gray sandy silt
	OCRPF-53	5 - 12	Gray clay
		12 - 36	Gray sandy silt
	OCRPF-66	3 - 5	Gray sandy silt
	OCRPF-77	8 - 18	Gray silty clay
Willow shrub	OCRPF-27	15 - 32	Gray sandy silt (Very dark brown)
	OCRPF-51	5 - 14	Gray clayey silt
	OCRPF-78	32 - 36	Gray sandy silt
Cattails/Willow shrub	OCRPF-122	3 - 36	Gray clay

Notes:

1. Samples include transects surveyed in 2010, and floodplain soil and sediment samples surveyed during 2011 Reconnaissance Work.

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Table 3 - Summary of Existing and Proposed Sample Densities by Strata

Proposed Sampling Strata	Unit	Area (acres)	Existing Sample Location Count	Existing Sample Location Density per Acre	Proposed Sample Location Count	Total Sample Location Count (Existing + Proposed)	Total Sample Location Density (Existing + Proposed) per Acre
Upland Area							
Upland Area-A	Upland Area 1	0.64	0	0.00	1	1	1.56
	Upland Area 2	3.80	1	0.26	1	2	0.53
	Upland Area 3	2.67	0	0.00	2	2	0.75
	Upland Area 4	6.08	1	0.16	3	4	0.66
	Upland Area (1-4)	13.19	2.00	0.15	7	9	0.68
Upland Area-B	Upland Area 5	0.80	0	0.00	1	1	1.25
	Upland Area 6	2.20	0	0.00	2	2	0.91
	Upland Area 7	5.33	2	0.38	1	3	0.56
	Upland Area 8	5.39	10	1.86	0	10	1.86
	Upland Area 9	6.28	1	0.16	3	4	0.64
	Upland Area 10	0.52	0	0.00	1	1	1.92
	Upland Area (5-10)	20.52	13.00	0.63	8	21	1.02
Floodplain Forest							
Floodplain Forest-A	Floodplain Forest 1	18.38	3	0.16	0	3	0.16
	Floodplain Forest 2	13.15	3	0.23	0	3	0.23
	Floodplain Forest 3	0.49	22	44.90	0	22	44.90
	Floodplain Forest - (1-3)	32.02	28.00	0.87	0	28	0.87
Floodplain Forest-B	Floodplain Forest 4	6.90	1	0.14	3	4	0.58
	Floodplain Forest 5	2.06	0	0.00	2	2	0.97
	Floodplain Forest 6	0.70	3	4.29	0	3	4.29
	Floodplain Forest 7a	5.30	3	0.57	5	8	1.51
	Floodplain Forest 7b	6.92	3	0.43	1	4	0.58
	Floodplain Forest - (4-7)	21.88	10.00	0.46	11	17	0.78
Low Terrace							
Low Terrace-A	Low Terrace 1	2.64	1	0.38	3	4	1.52
	Low Terrace 2	0.77	0	0.00	2	2	2.60
	Low Terrace 3	2.49	0	0.00	4	4	1.61
	Low Terrace (1-3)	5.90	1.00	0.17	9	10	1.69
Low Terrace-B	Low Terrace 4	6.94	4	0.58	7	11	1.59
	Low Terrace 5	1.22	0	0.00	2	2	1.64
	Low Terrace 6	3.06	3	0.98	2	5	1.63
	Low Terrace 7	1.89	0	0.00	3	3	1.59
	Low Terrace 8	6.41	2	0.31	8	10	1.56
	Low Terrace (4-8)	19.52	9.00	0.46	22	31	1.59
Low Terrace-C	Low Terrace 9	1.58	0	0.00	3	3	1.90
	Low Terrace 10	2.80	2	0.71	3	5	1.79
	Low Terrace 11	1.42	0	0.00	3	3	2.11
	Low Terrace 12	0.72	0	0.00	2	2	2.78
	Low Terrace (9-12)	6.52	2.00	0.31	11	13	1.99

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Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 3 - Summary of Existing and Proposed Sample Densities by Strata

Proposed Sampling Strata	Unit	Area (acres)	Existing Sample Location Count	Existing Sample Location Density per Acre	Proposed Sample Location Count	Total Sample Location Count (Existing + Proposed)	Total Sample Location Density (Existing + Proposed) per Acre
Emergent Wetland - Cattail							
Emergent Wetland - Cattail-A	Emergent Wetland 2 - Cattail	1.05	0	0.00	1	1	0.95
	Emergent Wetland 3 - Cattail	9.00	4	0.44	1	5	0.56
	Emergent Wetland 4 - Cattail	20.12	11	0.55	0	11	0.55
	Emergent Wetland 5 - Cattail	10.63	2	0.19	4	6	0.56
	Emergent Wetland - Cattail A (2-5)	40.80	17.00	0.42	6	23	0.56
Emergent Wetland - Cattail-B	Emergent Wetland 7 - Cattail	0.43	1	2.33	0	1	2.33
	Emergent Wetland 8 - Cattail	2.71	2	0.74	0	2	0.74
	Emergent Wetland 9 - Cattail	1.43	0	0.00	1	1	0.70
	Emergent Wetland - Cattail B (7-9)	4.57	3.00	0.66	1	4	0.88
Emergent Wetland - Purple Loosestrife (PLS)							
Emergent Wetland - PLS	Emergent Wetland 1 - PLS	1.80	5	2.78	0	5	2.78
	Emergent Wetland 6 - PLS	4.82	2	0.41	6	8	1.66
	Emergent Wetland - PLS (1,6)	6.62	7.00	1.06	6	13	1.96
Previous Channel							
Previous Channel A	Previous Channel 2	13.09	4	0.31	16	20	1.53
	Previous Channel 4	5.64	1	0.18	8	9	1.60
	Previous Channel 5	0.42	0	0.00	1	1	2.38
	Previous Channel (2,4,5)	19.15	5.00	0.26	25	30	1.57
Previous Channel B	Previous Channel 1	0.90	0	0.00	2	2	2.22
	Previous Channel 3	0.89	0	0.00	2	2	2.25
	Previous Channel (1,3)	1.79	0.00	0.00	4	4	2.23
Previous Channel C	Previous Channel 6	3.33	1	0.30	4	5	1.50
	Previous Channel 7	1.35	0	0.00	3	3	2.22
	Previous Channel 8	2.31	0	0.00	4	4	1.73
	Previous Channel 9	0.33	3	9.09	0	3	9.09
	Previous Channel 10	2.46	1	0.41	3	4	1.63
	Previous Channel (6-10)	9.78	5.00	0.51	14	19	1.94
Previous Channel D	Previous Channel 11	1.02	0	0.00	2	2	1.96
	Previous Channel 12	0.50	0	0.00	1	1	2.00
	Previous Channel 13	1.95	2	1.03	1	3	1.54
	Previous Channel 14	0.50	0	0.00	1	1	2.00
	Previous Channel 15	0.58	0	0.00	1	1	1.72
	Previous Channel 16	0.78	1	1.28	1	2	2.56
	Previous Channel 17	1.64	0	0.00	3	3	1.83
	Previous Channel (11-16)	6.97	3.00	0.43	10	13	1.87
Area between 700 ft elevation and SRI/FS boundary							
	Total Area	77.72	17	0.22	0	17	0.22
Total within SRI/FS boundary							
	Total Area	286.95	122	0.43	134	252	0.88

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Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 4 - Summary of Proposed Sediment and Soil Sampling Plan

Field Activity	Description	Number of Locations	Location IDs	Sample Intervals	Number of Samples	Analyses ^{4,5}
Soil Sampling	Bank Soil Sampling - Collect a soil core at the top of the bank at each bank profile.	68	OCTBS-1 through OCTBS-34 OCTBN-1 through OCTBN-34	0- to 6-inch, 6- to 12-inch, 12- to 24-inch, 12-inch increments deeper	286 ¹	PCB (all samples); particle size and TOC (surface only), TCL/TAL (select samples ⁴), dioxin/furan (select samples ⁴)
	Floodplain Soil Sampling - Sample to a density of 1.5 sample locations per acre in low lying areas and previous channels and 1 sample location per 2 acres in all other areas.	134	OCIFP-01 through OCIFP-134	0- to 6-inch, 6- to 12-inch, 12- to 24-inch, 12 inch increments deeper	563 ²	PCB (all samples); particle size and TOC (surface only), TCL/TAL (select samples ⁴), dioxin/furan (select samples ⁴)
Sediment	Sediment Sampling - Sample locations selected using a uniform random grid similar to former Plainwell Impoundment, with additional targeted sampling in the braided areas.	81	OCISED-01 to OCISED-81	0- to 2-inch, 2- to 6-inch, 6- to 12-inch, 12- to 24-inch, 12 inch increments deeper	511 ³	PCB (all cores); TOC and particle size (all surface and whole core for 20% of locations), TCL/TAL (select samples ⁴), dioxin/furan (select samples ⁴)
	Surface sediment re-sampling at select locations	22	OCISED-82 to OCISED-103	0- to 2-inch	22	PCB, TOC

Notes:

1. Bank soil samples based on an average 4 samples per core (average depth = 32 inches) plus 14 duplicates
2. Floodplain soil samples based on an average 4 samples per core (average depth = 32 inches) plus 27 duplicates
3. Sediment samples based on an average 6 samples per core (average depth of 4 feet) plus 25 duplicates
4. See Table 5 for proposed analyses
5. PCB - polychlorinated biphenyl
6. TOC - total organic carbon
7. TCL/TAL - Target Compound List / Target Analyte List

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Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Top-of-Bank	OCTBN-01	12,765,985.7	353,912.0	All	Surface	Surface				
Top-of-Bank	OCTBN-02	12,766,228.1	353,698.5	All	Surface	Surface				
Top-of-Bank	OCTBN-03	12,766,555.2	353,457.5	All	Surface	Surface	All	Archive	Archive	
Top-of-Bank	OCTBN-04	12,766,784.6	353,153.6	All	Surface	Surface				
Top-of-Bank	OCTBN-05	12,767,239.3	352,869.3	All	Surface	Surface				
Top-of-Bank	OCTBN-06	12,767,770.2	352,673.7	All	Surface	Surface		Archive	Archive	
Top-of-Bank	OCTBN-07	12,767,917.1	352,957.1	All	Surface	Surface				
Top-of-Bank	OCTBN-08	12,768,159.2	352,462.3	All	Surface	Surface				
Top-of-Bank	OCTBN-09	12,768,745.2	351,980.8	All	Surface	Surface				
Top-of-Bank	OCTBN-10	12,768,891.6	352,515.7	All	Surface	Surface				
Top-of-Bank	OCTBN-11	12,769,316.0	351,587.5	All	Surface	Surface	All	Archive	Archive	
Top-of-Bank	OCTBN-12	12,769,393.3	352,695.1	All	Surface	Surface				
Top-of-Bank	OCTBN-13	12,769,454.6	352,183.6	All	Surface	Surface				
Top-of-Bank	OCTBN-14	12,769,622.6	352,123.0	All	Surface	Surface				
Top-of-Bank	OCTBN-15	12,770,029.2	352,745.7	All	Surface	Surface				
Top-of-Bank	OCTBN-16	12,770,235.7	352,205.2	All	Surface	Surface		Archive	Archive	
Top-of-Bank	OCTBN-17	12,770,168.5	352,950.1	All	Surface	Surface				
Top-of-Bank	OCTBN-18	12,770,527.3	353,114.0	All	Surface	Surface				
Top-of-Bank	OCTBN-19	12,770,635.9	352,588.6	All	Surface	Surface				
Top-of-Bank	OCTBN-20	12,770,752.6	353,012.7	All	Surface	Surface				
Top-of-Bank	OCTBN-21	12,770,858.2	353,316.0	All	Surface	Surface	All	Archive	Archive	
Top-of-Bank	OCTBN-22	12,771,026.5	353,084.5	All	Surface	Surface				
Top-of-Bank	OCTBN-23	12,771,103.7	352,620.7	All	Surface	Surface				
Top-of-Bank	OCTBN-24	12,771,202.5	353,498.6	All	Surface	Surface				
Top-of-Bank	OCTBN-25	12,771,265.5	353,115.4	All	Surface	Surface				

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Top-of-Bank	OCTBN-26	12,771,452.5	353,972.7	All	Surface	Surface		Archive	Archive	
Top-of-Bank	OCTBN-27	12,771,630.0	352,948.4	All	Surface	Surface				
Top-of-Bank	OCTBN-28	12,771,711.4	353,239.2	All	Surface	Surface				
Top-of-Bank	OCTBN-29	12,771,700.7	353,940.6	All	Surface	Surface				
Top-of-Bank	OCTBN-30	12,771,866.3	353,508.7	All	Surface	Surface				
Top-of-Bank	OCTBN-31	12,772,076.3	353,648.5	All	Surface	Surface	All	Archive	Archive	
Top-of-Bank	OCTBN-32	12,772,163.4	353,003.5	All	Surface	Surface				
Top-of-Bank	OCTBN-33	12,772,201.5	352,082.4	All	Surface	Surface				
Top-of-Bank	OCTBN-34	12,772,122.2	351,684.0	All	Surface	Surface				
Top-of-Bank	OCTBS-01	12,765,798.0	353,631.6	All	Surface	Surface				
Top-of-Bank	OCTBS-02	12,766,093.9	353,449.0	All	Surface	Surface		Archive	Archive	
Top-of-Bank	OCTBS-03	12,766,368.2	353,279.8	All	Surface	Surface				
Top-of-Bank	OCTBS-04	12,766,604.3	352,952.9	All	Surface	Surface				
Top-of-Bank	OCTBS-05	12,767,126.9	352,581.2	All	Surface	Surface				
Top-of-Bank	OCTBS-06	12,767,645.8	352,427.0	All	Surface	Surface				
Top-of-Bank	OCTBS-07	12,767,913.5	352,877.6	All	Surface	Surface	All	Archive	Archive	
Top-of-Bank	OCTBS-08	12,768,125.1	352,196.7	All	Surface	Surface				
Top-of-Bank	OCTBS-09	12,768,644.6	351,961.4	All	Surface	Surface				
Top-of-Bank	OCTBS-10	12,768,945.9	352,278.5	All	Surface	Surface				
Top-of-Bank	OCTBS-11	12,769,313.1	351,513.1	All	Surface	Surface				
Top-of-Bank	OCTBS-12	12,769,474.2	352,509.8	All	Surface	Surface				
Top-of-Bank	OCTBS-13	12,769,454.3	352,145.2	All	Surface	Surface				
Top-of-Bank	OCTBS-14	12,769,673.3	352,109.5	All	Surface	Surface				
Top-of-Bank	OCTBS-15	12,770,009.5	352,563.9	All	Surface	Surface		Archive	Archive	
Top-of-Bank	OCTBS-16	12,770,363.3	352,107.7	All	Surface	Surface				

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Top-of-Bank	OCTBS-17	12,770,227.5	352,889.3	All	Surface	Surface	All	Archive	Archive	
Top-of-Bank	OCTBS-18	12,770,533.2	353,030.7	All	Surface	Surface				
Top-of-Bank	OCTBS-19	12,770,623.0	352,425.2	All	Surface	Surface				
Top-of-Bank	OCTBS-20	12,770,736.4	352,950.9	All	Surface	Surface				
Top-of-Bank	OCTBS-21	12,770,894.4	353,294.1	All	Surface	Surface				
Top-of-Bank	OCTBS-22	12,771,061.4	353,022.0	All	Surface	Surface		Archive	Archive	
Top-of-Bank	OCTBS-23	12,771,183.7	352,496.3	All	Surface	Surface				
Top-of-Bank	OCTBS-24	12,771,271.2	353,499.2	All	Surface	Surface				
Top-of-Bank	OCTBS-25	12,771,354.7	353,046.0	All	Surface	Surface				
Top-of-Bank	OCTBS-26	12,771,478.6	353,920.6	All	Surface	Surface				
Top-of-Bank	OCTBS-27	12,771,627.4	352,814.4	All	Surface	Surface	All	Archive	Archive	
Top-of-Bank	OCTBS-28	12,771,698.6	353,133.4	All	Surface	Surface				
Top-of-Bank	OCTBS-29	12,771,653.7	353,897.4	All	Surface	Surface				
Top-of-Bank	OCTBS-30	12,771,857.4	353,497.7	All	Surface	Surface				
Top-of-Bank	OCTBS-31	12,772,014.6	353,605.6	All	Surface	Surface				
Top-of-Bank	OCTBS-32	12,772,068.8	352,937.5	All	Surface	Surface		Archive	Archive	
Top-of-Bank	OCTBS-33	12,772,054.7	352,129.1	All	Surface	Surface				
Top-of-Bank	OCTBS-34	12,771,972.2	351,700.1	All	Surface	Surface				
Floodplain Soil	OCIFP-01	12,765,761.1	353,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-02	12,765,861.1	353,543.6	All	Surface	Surface				
Floodplain Soil	OCIFP-03	12,765,961.1	353,943.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-04	12,766,161.1	353,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-05	12,766,361.1	353,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-06	12,766,561.1	353,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-07	12,766,661.1	353,543.6	All	Surface	Surface				

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Floodplain Soil	OCIFP-08	12,766,761.1	353,543.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-09	12,766,861.1	353,143.6	All	Surface	Surface				
Floodplain Soil	OCIFP-10	12,766,961.1	353,243.6	All	Surface	Surface				
Floodplain Soil	OCIFP-11	12,766,961.1	352,416.6	All	Surface	Surface				
Floodplain Soil	OCIFP-12	12,767,061.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-13	12,767,161.1	353,443.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-14	12,767,161.1	353,243.6	All	Surface	Surface				
Floodplain Soil	OCIFP-15	12,767,161.1	352,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-16	12,767,161.1	352,443.6	All	Surface	Surface				
Floodplain Soil	OCIFP-17	12,767,261.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-18	12,767,561.1	353,043.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-19	12,767,561.1	352,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-20	12,767,561.1	352,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-21	12,767,561.1	352,143.6	All	Surface	Surface				
Floodplain Soil	OCIFP-22	12,767,661.1	352,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-23	12,767,861.1	353,043.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-24	12,767,861.1	352,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-25	12,767,961.1	352,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-26	12,767,961.1	352,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-27	12,768,061.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-28	12,768,061.1	352,843.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-29	12,768,061.1	352,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-30	12,768,061.1	352,543.6	All	Surface	Surface				
Floodplain Soil	OCIFP-31	12,768,161.1	352,543.6	All	Surface	Surface				
Floodplain Soil	OCIFP-32	12,768,161.1	351,943.6	All	Surface	Surface				

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Floodplain Soil	OCIFP-33	12,768,261.1	352,843.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-34	12,768,261.1	352,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-35	12,768,361.1	351,443.6	All	Surface	Surface				
Floodplain Soil	OCIFP-36	12,768,461.1	352,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-37	12,768,461.1	352,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-38	12,768,461.1	352,643.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-39	12,768,461.1	351,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-40	12,768,561.1	352,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-41	12,768,561.1	352,543.6	All	Surface	Surface				
Floodplain Soil	OCIFP-42	12,768,561.1	351,543.6	All	Surface	Surface				
Floodplain Soil	OCIFP-43	12,768,661.1	352,543.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-44	12,768,661.1	351,343.6	All	Surface	Surface				
Floodplain Soil	OCIFP-45	12,768,661.1	351,243.6	All	Surface	Surface				
Floodplain Soil	OCIFP-46	12,768,761.1	352,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-47	12,768,761.1	351,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-48	12,768,761.1	351,443.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-49	12,768,861.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-50	12,768,861.1	352,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-51	12,768,861.1	351,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-52	12,768,861.1	351,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-53	12,768,961.1	353,143.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-54	12,769,061.1	352,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-55	12,769,061.1	351,343.6	All	Surface	Surface				
Floodplain Soil	OCIFP-56	12,769,161.1	352,643.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-57	12,769,161.1	352,343.6	All	Surface	Surface				

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Floodplain Soil	OCIFP-58	12,769,161.1	352,243.6	All	Surface	Surface	All	Archive	Archive	Yes
Floodplain Soil	OCIFP-59	12,769,161.1	352,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-60	12,769,161.1	351,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-61	12,769,161.1	351,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-62	12,769,261.1	352,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-63	12,769,261.1	352,243.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-64	12,769,261.1	351,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-65	12,769,261.1	351,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-66	12,769,361.1	352,843.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-67	12,769,361.1	352,443.6	All	Surface	Surface				
Floodplain Soil	OCIFP-68	12,769,461.1	352,443.6	All	Surface	Surface	All	Archive	Archive	Yes
Floodplain Soil	OCIFP-69	12,769,461.1	352,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-70	12,769,661.1	352,443.6	All	Surface	Surface				
Floodplain Soil	OCIFP-71	12,769,661.1	352,243.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-72	12,769,661.1	351,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-73	12,769,761.1	352,443.6	All	Surface	Surface		Archive	Archive	Yes
Floodplain Soil	OCIFP-74	12,769,761.1	352,143.6	All	Surface	Surface				
Floodplain Soil	OCIFP-75	12,769,761.1	352,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-76	12,769,861.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-77	12,769,861.1	352,943.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-78	12,769,861.1	352,843.6	All	Surface	Surface	All	Archive	Archive	Yes
Floodplain Soil	OCIFP-79	12,769,861.1	352,443.6	All	Surface	Surface				
Floodplain Soil	OCIFP-80	12,769,961.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-81	12,769,961.1	352,543.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-82	12,770,061.1	352,943.6	All	Surface	Surface				Yes

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Floodplain Soil	OCIFP-83	12,770,061.1	352,743.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-84	12,770,161.1	352,343.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-85	12,770,161.1	352,243.6	All	Surface	Surface				
Floodplain Soil	OCIFP-86	12,770,261.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-87	12,770,361.1	352,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-88	12,770,461.1	352,843.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-89	12,770,461.1	352,743.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-90	12,770,461.1	352,143.6	All	Surface	Surface				
Floodplain Soil	OCIFP-91	12,770,561.1	352,943.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-92	12,770,561.1	352,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-93	12,770,761.1	351,843.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-94	12,770,861.1	353,143.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-95	12,770,861.1	351,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-96	12,770,961.1	353,143.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-97	12,770,961.1	352,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-98	12,771,061.1	353,443.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-99	12,771,061.1	353,343.6	All	Surface	Surface				
Floodplain Soil	OCIFP-100	12,771,061.1	353,243.6	All	Surface	Surface				
Floodplain Soil	OCIFP-101	12,771,061.1	352,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-102	12,771,161.1	353,543.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-103	12,771,161.1	352,143.6	All	Surface	Surface				
Floodplain Soil	OCIFP-104	12,771,261.1	353,243.6	All	Surface	Surface				
Floodplain Soil	OCIFP-105	12,771,261.1	351,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-106	12,771,361.1	352,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-107	12,771,361.1	352,643.6	All	Surface	Surface		Archive	Archive	

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Floodplain Soil	OCIFP-108	12,771,361.1	352,343.6	All	Surface	Surface				
Floodplain Soil	OCIFP-109	12,771,461.1	353,843.6	All	Surface	Surface				
Floodplain Soil	OCIFP-110	12,771,461.1	352,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-111	12,771,461.1	352,543.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-112	12,771,561.1	353,443.6	All	Surface	Surface				
Floodplain Soil	OCIFP-113	12,771,561.1	352,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-114	12,771,661.1	353,843.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-115	12,771,661.1	352,743.6	All	Surface	Surface				
Floodplain Soil	OCIFP-116	12,771,761.1	353,943.6	All	Surface	Surface		Archive	Archive	Yes
Floodplain Soil	OCIFP-117	12,771,761.1	353,343.6	All	Surface	Surface				
Floodplain Soil	OCIFP-118	12,771,761.1	352,543.6	All	Surface	Surface				
Floodplain Soil	OCIFP-119	12,771,861.1	354,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-120	12,771,861.1	352,243.6	All	Surface	Surface	All	Archive	Archive	
Floodplain Soil	OCIFP-121	12,772,061.1	353,943.6	All	Surface	Surface				
Floodplain Soil	OCIFP-122	12,772,061.1	353,143.6	All	Surface	Surface				
Floodplain Soil	OCIFP-123	12,772,061.1	352,343.6	All	Surface	Surface				
Floodplain Soil	OCIFP-124	12,772,161.1	353,443.6	All	Surface	Surface				Yes
Floodplain Soil	OCIFP-125	12,772,161.1	353,143.6	All	Surface	Surface		Archive	Archive	
Floodplain Soil	OCIFP-126	12,772,261.1	353,643.6	All	Surface	Surface				
Floodplain Soil	OCIFP-127	12,772,361.1	353,543.6	All	Surface	Surface				
Floodplain Soil	OCIFP-128	12,772,361.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-129	12,772,461.1	353,343.6	All	Surface	Surface				
Floodplain Soil	OCIFP-130	12,772,461.1	353,143.6	All	Surface	Surface				
Floodplain Soil	OCIFP-131	12,772,561.1	353,043.6	All	Surface	Surface				
Floodplain Soil	OCIFP-132	12,772,561.1	352,843.6	All	Surface	Surface				Yes

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Floodplain Soil	OCIFP-133	12,772,661.1	353,343.6	All	Surface	Surface				
Floodplain Soil	OCIFP-134	12,772,761.1	352,643.6	All	Surface	Surface		Archive	Archive	Yes
Sediment	OCISED-01	12,765,900.0	353,702.8	All	Surface	Surface				
Sediment	OCISED-02	12,765,940.2	353,891.3	All	Surface	Surface				
Sediment	OCISED-03	12,765,972.1	353,607.8	All	Surface	Surface	All	Archive	Archive	
Sediment	OCISED-04	12,766,192.1	353,487.8	All	Surface	Surface				
Sediment	OCISED-05	12,766,297.5	353,563.7	All	Surface	Surface				
Sediment	OCISED-06	12,766,362.7	353,299.1	All	Surface	Surface				
Sediment	OCISED-07	12,766,410.0	353,489.5	All	Surface	Surface				
Sediment	OCISED-08	12,766,598.4	352,972.1	All	Surface	Surface		Archive	Archive	
Sediment	OCISED-09	12,766,602.5	353,151.6	All	Surface	Surface				
Sediment	OCISED-10	12,766,647.9	353,046.2	All	Surface	Surface				
Sediment	OCISED-11	12,766,692.5	353,698.8	All	Surface	Surface				
Sediment	OCISED-12	12,766,732.1	352,707.9	All	Surface	Surface				
Sediment	OCISED-13	12,766,739.2	353,132.8	All	Surface	Surface	All	Archive	Archive	
Sediment	OCISED-14	12,766,812.1	352,827.9	All	Surface	Surface				
Sediment	OCISED-15	12,766,872.1	352,987.9	All	Surface	Surface				
Sediment	OCISED-16	12,766,909.1	352,803.6	All	Surface	Surface				
Sediment	OCISED-17	12,767,652.1	352,567.9	All	Surface	Surface				
Sediment	OCISED-18	12,767,672.6	352,757.4	All	Surface	Surface		Archive	Archive	
Sediment	OCISED-19	12,767,852.1	352,327.9	All	Surface	Surface				
Sediment	OCISED-20	12,767,926.5	352,894.4	All	Surface	Surface				
Sediment	OCISED-21	12,768,102.4	352,216.1	All	Surface	Surface				
Sediment	OCISED-22	12,768,132.0	352,441.4	All	Surface	Surface				
Sediment	OCISED-23	12,768,246.9	352,356.2	All	Surface	Surface	All	Archive	Archive	

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Sediment	OCISED-24	12,768,304.8	353,071.9	All	Surface	Surface				
Sediment	OCISED-25	12,768,442.8	352,237.1	All	Surface	Surface				
Sediment	OCISED-26	12,768,515.8	353,077.1	All	Surface	Surface				
Sediment	OCISED-27	12,768,686.9	352,737.4	All	Surface	Surface				
Sediment	OCISED-28	12,768,689.5	351,851.7	All	Surface	Surface		Archive	Archive	
Sediment	OCISED-29	12,768,694.6	352,114.2	All	Surface	Surface				
Sediment	OCISED-30	12,768,721.2	351,926.8	All	Surface	Surface				
Sediment	OCISED-31	12,769,109.3	352,115.2	All	Surface	Surface				
Sediment	OCISED-32	12,769,183.5	351,559.9	All	Surface	Surface				
Sediment	OCISED-33	12,769,331.6	352,566.6	All	Surface	Surface	All	Archive	Archive	
Sediment	OCISED-34	12,769,369.8	352,674.6	All	Surface	Surface				
Sediment	OCISED-35	12,769,661.3	352,868.9	All	Surface	Surface				
Sediment	OCISED-36	12,769,678.2	352,631.6	All	Surface	Surface				
Sediment	OCISED-37	12,769,780.0	351,808.2	All	Surface	Surface				
Sediment	OCISED-38	12,769,801.2	352,713.9	All	Surface	Surface		Archive	Archive	
Sediment	OCISED-39	12,769,839.7	353,072.4	All	Surface	Surface				
Sediment	OCISED-40	12,769,985.2	352,583.3	All	Surface	Surface				
Sediment	OCISED-41	12,770,188.2	352,930.6	All	Surface	Surface				
Sediment	OCISED-42	12,770,277.2	353,050.0	All	Surface	Surface				
Sediment	OCISED-43	12,770,436.7	352,257.7	All	Surface	Surface	All	Archive	Archive	
Sediment	OCISED-44	12,770,561.5	353,115.5	All	Surface	Surface				
Sediment	OCISED-45	12,770,655.4	352,602.5	All	Surface	Surface				
Sediment	OCISED-46	12,770,784.8	353,018.0	All	Surface	Surface				
Sediment	OCISED-47	12,770,789.2	353,235.6	All	Surface	Surface				
Sediment	OCISED-48	12,771,188.8	353,032.0	All	Surface	Surface		Archive	Archive	

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Sediment	OCISED-49	12,771,232.1	353,590.0	All	Surface	Surface				
Sediment	OCISED-50	12,771,274.5	352,648.4	All	Surface	Surface				
Sediment	OCISED-51	12,771,314.4	352,515.7	All	Surface	Surface				
Sediment	OCISED-52	12,771,434.0	352,849.0	All	Surface	Surface				
Sediment	OCISED-53	12,771,436.3	353,120.1	All	Surface	Surface	All	Archive	Archive	
Sediment	OCISED-54	12,771,492.3	353,956.3	All	Surface	Surface				
Sediment	OCISED-55	12,771,645.9	352,902.3	All	Surface	Surface				
Sediment	OCISED-56	12,771,824.6	353,906.1	All	Surface	Surface				
Sediment	OCISED-57	12,771,894.1	353,419.5	All	Surface	Surface				
Sediment	OCISED-58	12,771,897.1	352,444.1	All	Surface	Surface		Archive	Archive	
Sediment	OCISED-59	12,772,026.0	353,189.5	All	Surface	Surface				
Sediment	OCISED-60	12,772,044.1	353,633.0	All	Surface	Surface				
Sediment	OCISED-61	12,772,217.7	353,142.0	All	Surface	Surface				
Sediment	OCISED-62	12,772,232.0	351,243.3	All	Surface	Surface				
Sediment	OCISED-63	12,772,252.5	351,794.5	All	Surface	Surface	All	Archive	Archive	
Sediment	OCISED-64	12,772,260.6	353,591.3	All	Surface	Surface				
Sediment	OCISED-65	12,772,300.5	353,376.6	All	Surface	Surface				
Sediment	OCISED-66	12,772,315.8	353,775.3	All	Surface	Surface				
Sediment	OCISED-67	12,772,321.3	352,208.3	All	Surface	Surface				
Sediment	OCISED-68	12,772,338.2	351,939.4	All	Surface	Surface		Archive	Archive	
Sediment	OCISED-69	12,772,354.6	352,658.9	All	Surface	Surface				
Sediment	OCISED-70	12,772,373.6	351,291.5	All	Surface	Surface				
Sediment	OCISED-71	12,772,399.9	351,204.5	All	Surface	Surface				
Sediment	OCISED-72	12,772,415.4	351,638.9	All	Surface	Surface				
Sediment	OCISED-73	12,772,493.7	353,852.0	All	Surface	Surface	All	Archive	Archive	

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

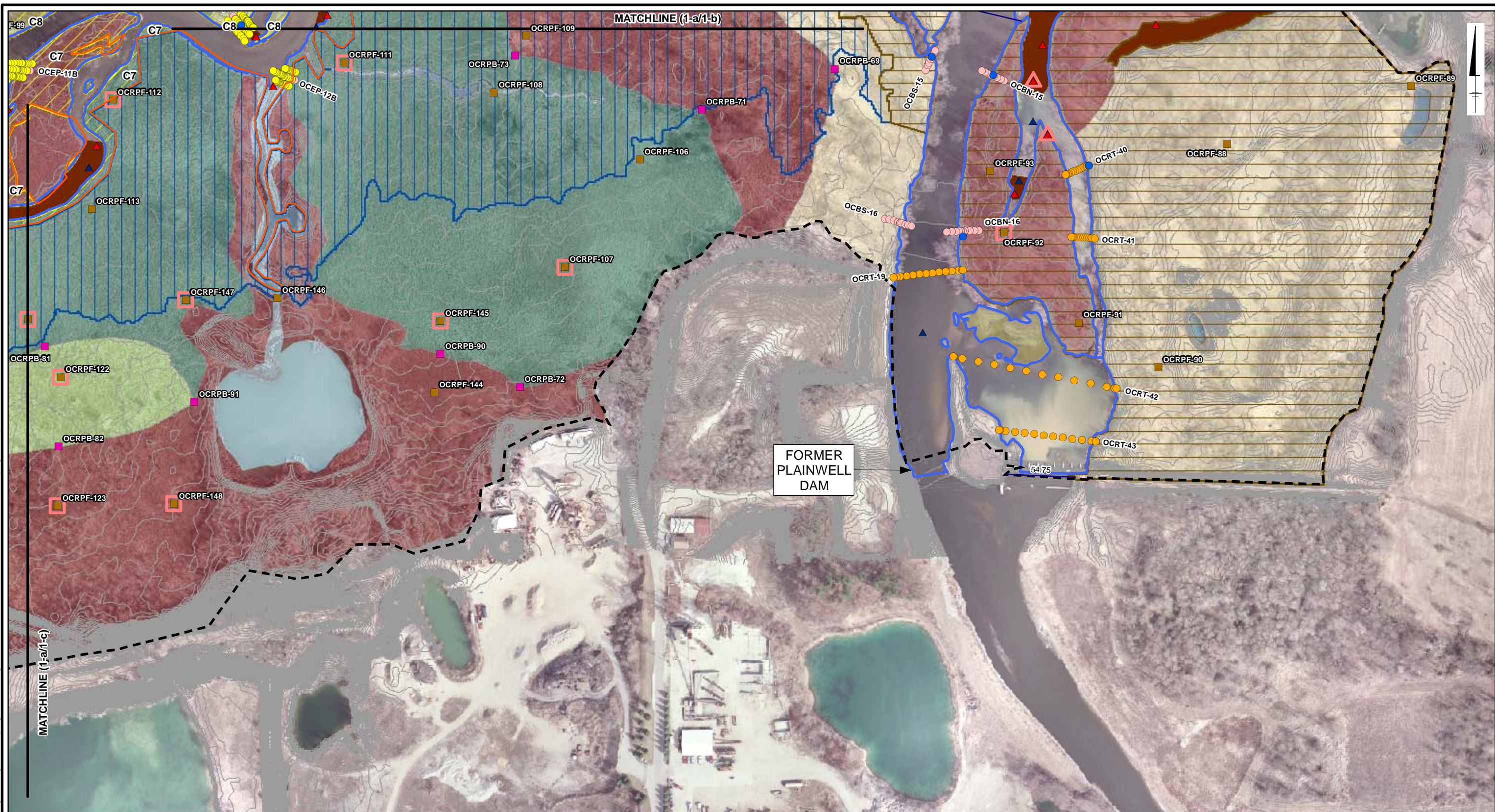
Table 5 - Non-PCB and Deeper Sample Analysis

Sample Type	Location	Easting	Northing	Samples for Analyses						Target 60-inch Depth
				PCB Aroclor	TOC	Grain Size	VOCs/ Mercury	SVOCs/ Pesticides/ Metals	Dioxin/furan	
Sediment	OCISED-74	12,772,640.7	352,188.2	All	Surface	Surface				
Sediment	OCISED-75	12,772,705.3	353,741.8	All	Surface	Surface				
Sediment	OCISED-76	12,772,733.0	353,239.8	All	Surface	Surface				
Sediment	OCISED-77	12,772,801.4	352,228.9	All	Surface	Surface				
Sediment	OCISED-78	12,772,831.1	352,596.3	All	Surface	Surface		Archive	Archive	
Sediment	OCISED-79	12,772,875.7	352,776.2	All	Surface	Surface				
Sediment	OCISED-80	12,772,953.8	352,654.5	All	Surface	Surface				
Sediment	OCISED-81	12,772,991.2	352,319.0	All	Surface	Surface				

Notes:

All All samples in core
Surface Surface samples only
Archive Archive all samples/splits for potential future analyses

Figures



NOTES:

1. AERIAL IMAGE DERIVED FROM ORTHOGRAPHIC DATA BY AXIS GEOSPATIAL, LLC. OTSEGO CITY AREA FLOWN SPRING 2010.

2. SURVEY DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN WINTER 2010/2011. RECONNAISSANCE DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN MAY 2011.

LEGEND:

EXISTING GEOMORPHIC CLASSIFICATIONS PER RECONNAISSANCE PLAN:

- PREVIOUS MAIN CHANNEL
- PREVIOUS CHANNEL
- LOWER TERRACE
- MEDIUM TERRACE
- UPPER TERRACE
- ADDITIONAL RECONNAISSANCE AREA
- SEDIMENT DEPOSITIONAL AREA IDENTIFIED DURING AREA RECONNAISSANCE
- SRI/FS STUDY AREA (CH2M HILL 2003)

SEDIMENT BOUNDARY RECON LOCATION

SEDIMENT DEPOSITION AREA RECON LOCATION

BANK PROFILE

DAM SURVEY

EROSION PIN PROFILE

RIVER TRANSECT

USGS TRANSECT MARKER POST LOCATION (LOCATION VERIFIED IN 2010/2011 SURVEY)

FLOODPLAIN SOIL RECON LOCATION

GEOMORPHIC BOUNDARY RECON LOCATION

CHANNEL AREA

FLOODPLAIN SOIL RECON LOCATION WHERE GRAY CLAY OR SILT WAS OBSERVED

RIVER TRANSECT LOCATIONS WHERE CLAY OR SILT WAS OBSERVED

SEDIMENT RECON LOCATION WHERE GRAY CLAY OR SILT WAS OBSERVED

VEGETATION TYPE:

- WETLAND MEADOW COMPLEX
- FLOODPLAIN FOREST - ASPEN
- CATTAILS/WILLOW SHRUB

PURPLE LOOSE STRIFE

CATTAILS

FLOODPLAIN FOREST

WATER

PHRAGMITES

REED CANARY GRASS

UPLAND FOREST

WILLOW SHRUB

RIVER MILES

EXTENT OF CURRENT IMPOUNDMENT

0 250 500 Feet

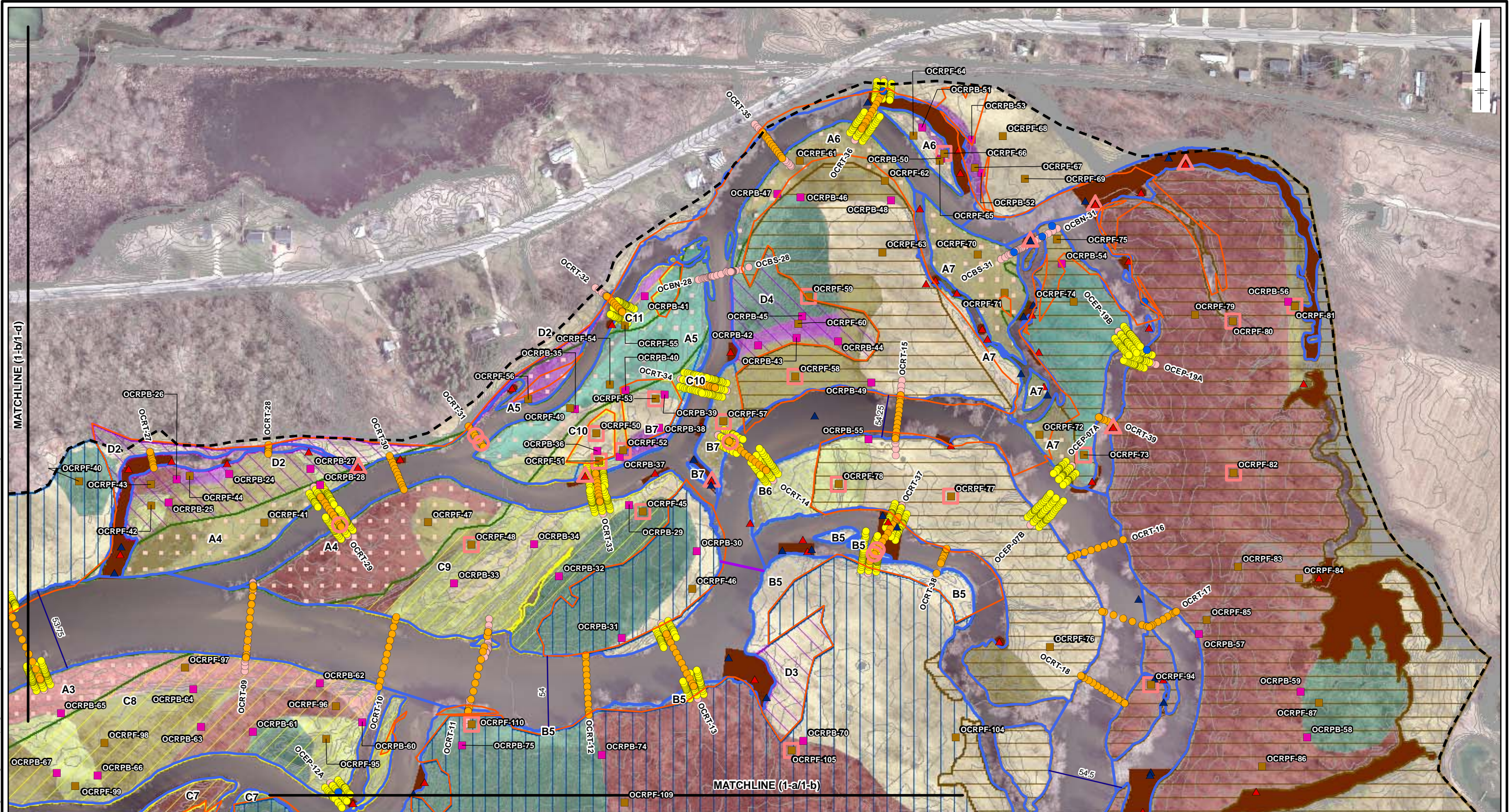
GRAPHIC SCALE

GEORGIA-PACIFIC LLC
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE
**AREA 2 SOIL AND SEDIMENT
FIELD SAMPLING PLAN**

**IDENTIFICATION OF FLOODPLAIN
AREAS BY VEGETATION TYPE**

ARCADIS

**FIGURE
1-a**



NOTES:

1. AERIAL IMAGE DERIVED FROM ORTHOGRAPHIC DATA BY AXIS GEOSPATIAL, LLC. OTSEGO CITY AREA FLOWN SPRING 2010.
2. SURVEY DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN WINTER 2010/2011. RECONNAISSANCE DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN MAY 2011.

0 250 500 Feet
GRAPHIC SCALE

LEGEND:

EXISTING GEOMORPHIC CLASSIFICATIONS PER RECONNAISSANCE PLAN:

- PREVIOUS MAIN CHANNEL
- PREVIOUS CHANNEL
- LOWER TERRACE
- MEDIUM TERRACE
- UPPER TERRACE
- ADDITIONAL RECONNAISSANCE AREA
- SEDIMENT DEPOSITIONAL AREA IDENTIFIED DURING AREA RECONNAISSANCE
- SRI/FS STUDY AREA (CH2M HILL 2003)

- SEDIMENT BOUNDARY RECON LOCATION
- SEDIMENT DEPOSITION AREA RECON LOCATION
- BANK PROFILE
- DAM SURVEY
- EROSION PIN PROFILE
- RIVER TRANSECT
- USGS TRANSECT MARKER POST LOCATION (LOCATION VERIFIED IN 2010/2011 SURVEY)
- FLOODPLAIN SOIL RECON LOCATION
- GEOMORPHIC BOUNDARY RECON LOCATION
- CHANNEL AREA

FLOODPLAIN SOIL RECON LOCATION WHERE GRAY CLAY OR SILT WAS OBSERVED

RIVER TRANSECT LOCATIONS WHERE CLAY OR SILT WAS OBSERVED

SEDIMENT RECON LOCATION WHERE GRAY CLAY OR SILT WAS OBSERVED

VEGETATION TYPE:

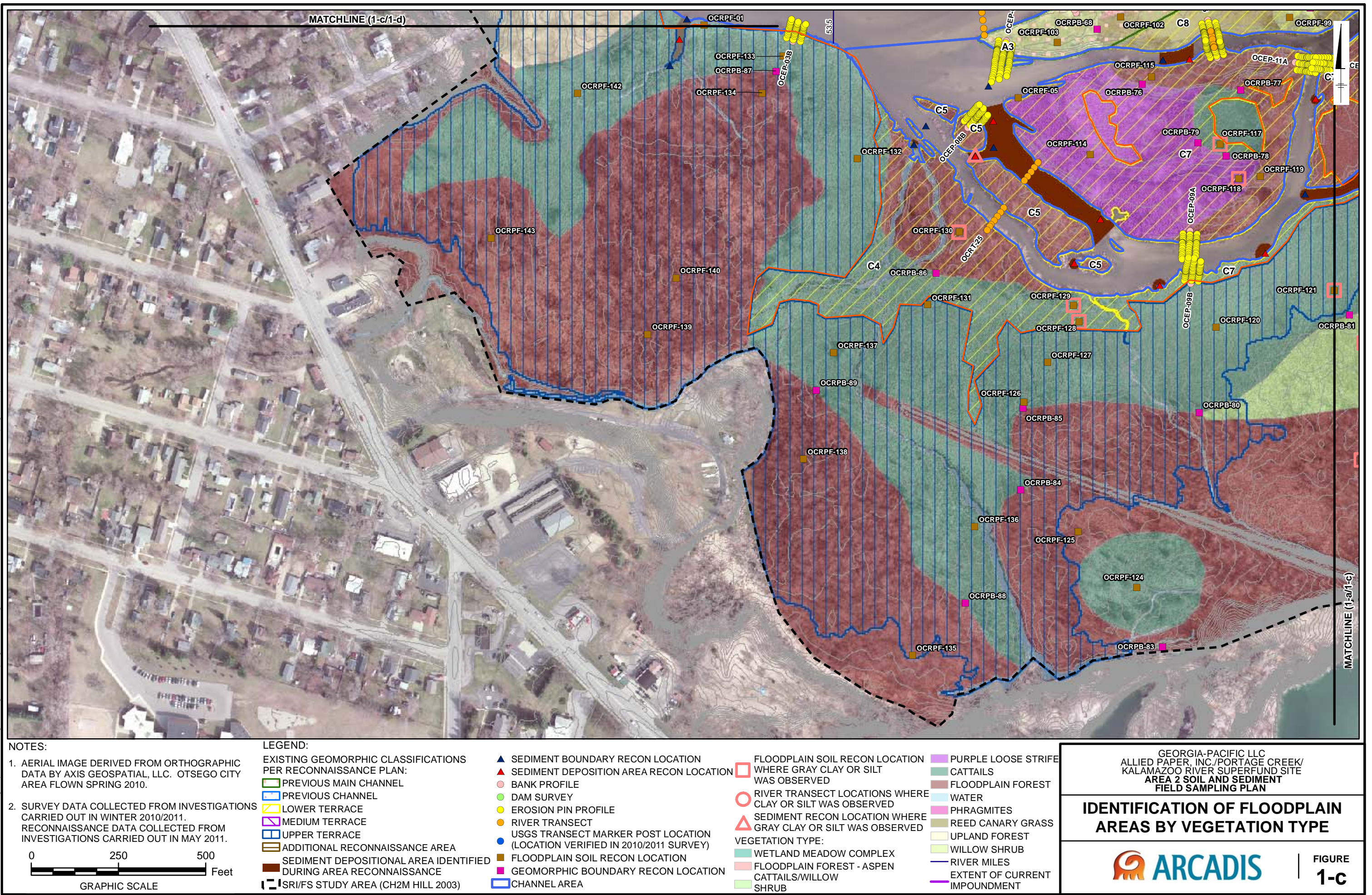
- WETLAND MEADOW COMPLEX
- FLOODPLAIN FOREST - ASPEN CATTAILS/WILLOW SHRUB
- PURPLE LOOSE STRIFE
- CATTAILS
- FLOODPLAIN FOREST
- WATER
- PHRAGMITES
- REED CANARY GRASS
- UPLAND FOREST
- WILLOW SHRUB
- RIVER MILES
- EXTENT OF CURRENT IMPOUNDMENT

GEORGIA-PACIFIC LLC
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE
AREA 2 SOIL AND SEDIMENT
FIELD SAMPLING PLAN

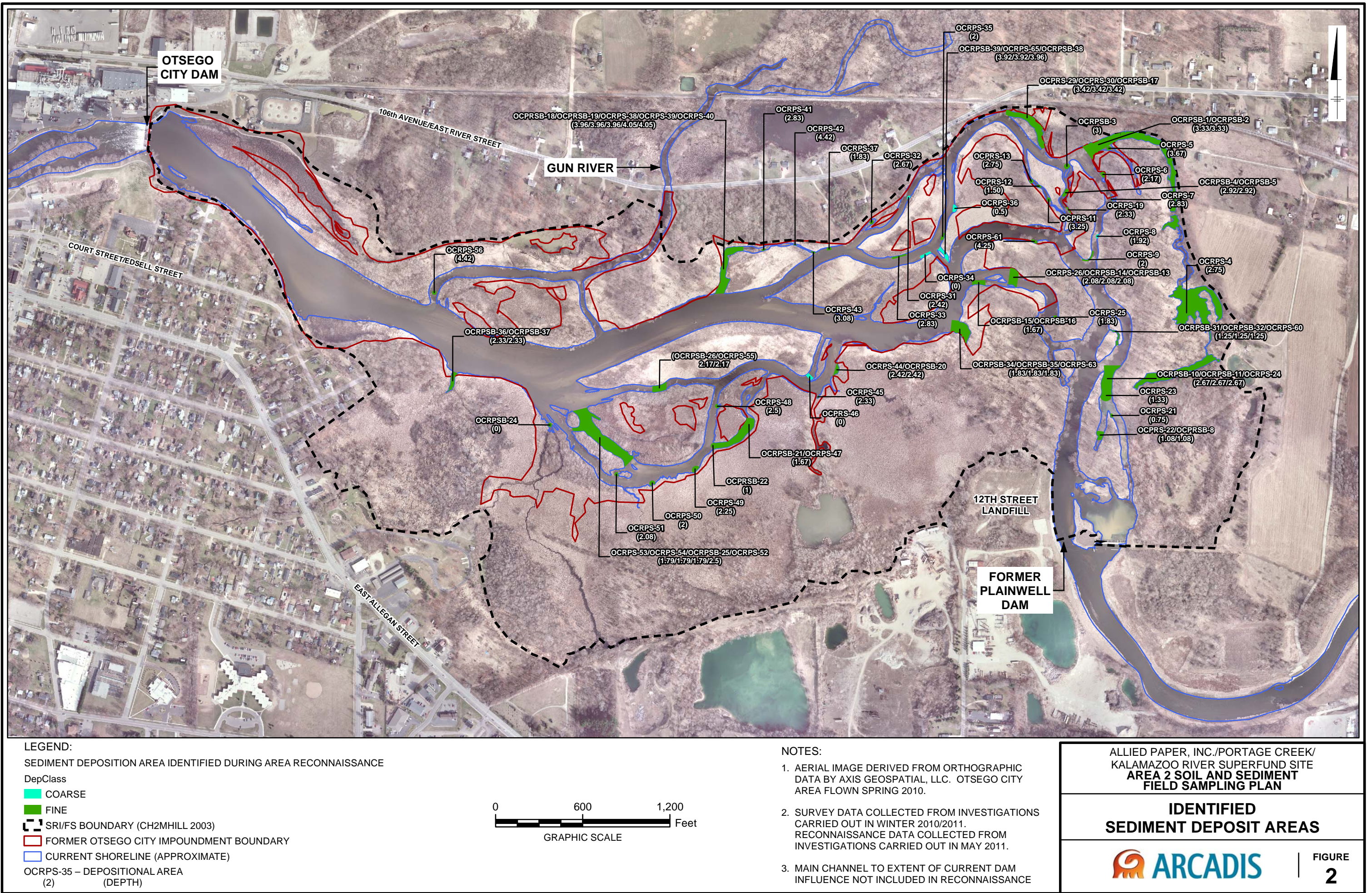
**IDENTIFICATION OF FLOODPLAIN
AREAS BY VEGETATION TYPE**

ARCADIS

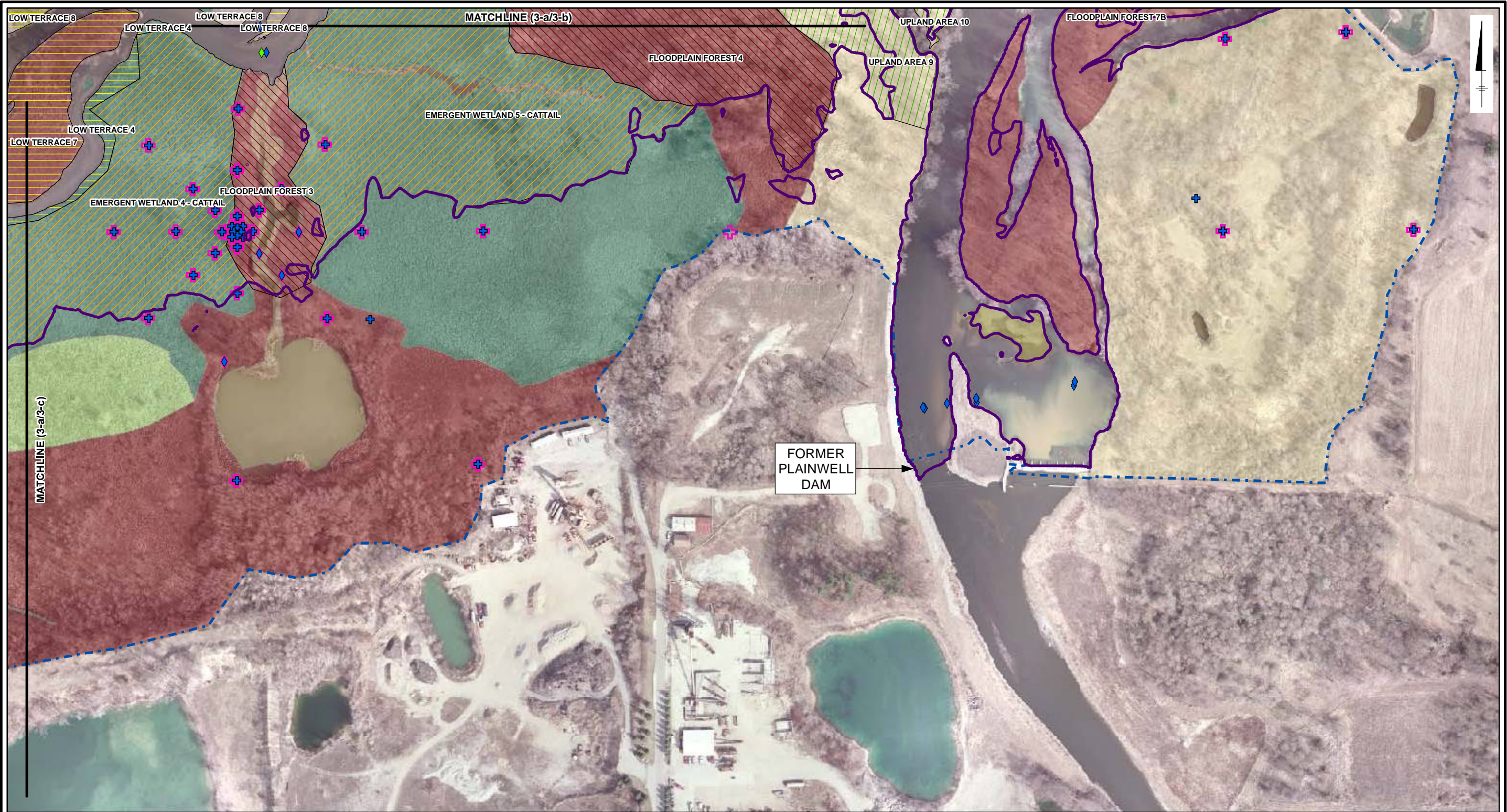
FIGURE
1-b



City: SYR Div/Group: 90 Created By: Sruti Pulugurtha Last Saved By: spulugurtha
KRSB - Area 2 Field Sampling Plan (B0064529.0002.00500)
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KRSO - Otsego City (B0064529.0002.00500)
Q:\KRSO\PlainwellDamToOtsegoCityDam\PreDesignInvestigation\SamplingPlan\mxd\Internal - Floodplain Sampling Strata Review_v3.mxd 11/7/2011 12:55:56 PM (Page 1 of 4)



LEGEND:

- EMERGENT WETLAND
- FLOODPLAIN FOREST
- LOW TERRACE
- PREVIOUS CHANNEL
- UPLAND AREA
- 700 FT CONTOUR

VEGETATION TYPE:

- WETLAND MEADOW COMPLEX
- FLOODPLAIN FOREST - ASPEN

- CATTAILS/WILLOW SHRUB
- CATTAILS
- FLOODPLAIN FOREST
- OPEN WATER
- PHRAGMITES
- PURPLE LOOSE STRIFE
- REED CANARY GRASS
- UPLAND FOREST
- WILLOW SHRUB

EXISTING SEDIMENT PCB SAMPLE LOCATION

MAXIMUM PCB RESULTS (MG/KG):

- ND - 1
- 1 - 5
- 5 - 10
- 10 - 50
- > 50

EXTENT OF CURRENT IMPOUNDMENT (APPROXIMATE - SEE NOTE 1)

- EPA SEDIMENT SAMPLE LOCATION

EPA SOIL SAMPLE LOCATION

EXISTING SOIL PCB SAMPLE LOCATION

- MAXIMUM PCB RESULTS (MG/KG):
- ND - 1
- 1 - 5
- 5 - 10
- 10 - 50
- > 50

- SRI/FS STUDY AREA (CH2M HILL 2003)

NOTES:

- EXTENT OF CURRENT IMPOUNDMENT WAS REPORTED TO BE LOCATED AT USGS TRANSECT AT G10 IN WATER RESOURCE INVESTIGATIONS REPORT 03-4218 (USGS 2004).
- AERIAL IMAGE DERIVED FROM ORTHOGRAPHIC DATA BY AXIS GEOSPATIAL, LLC. OTSEGO CITY AREA FLOWN SPRING 2010.
- DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN WINTER 2010/2011.
- SAMPLE SLO55 EXCLUDED AS IT FALLS WITHIN THE BOUNDARY OF THE CLEAN UP AREA AT OU-4.

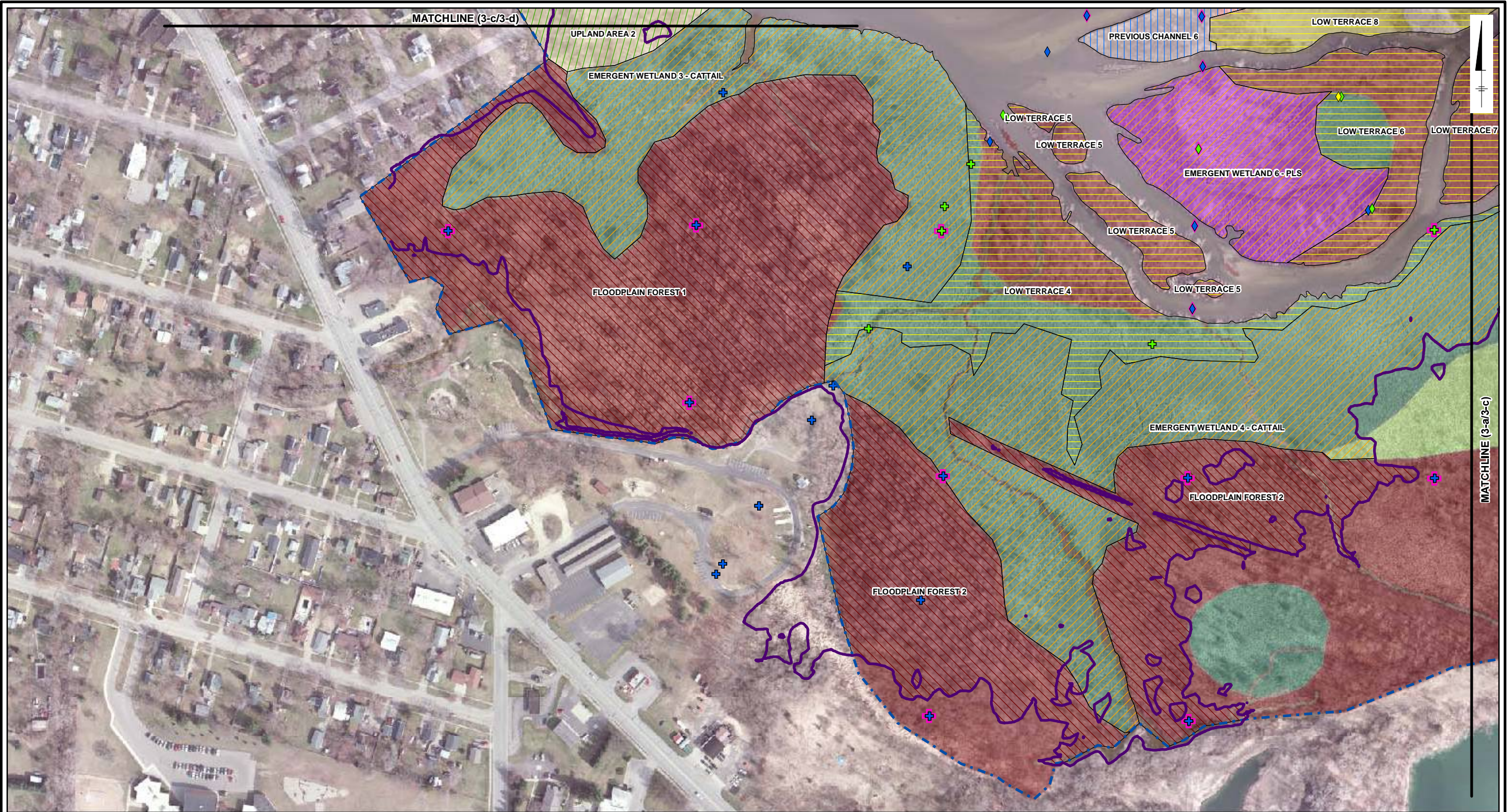


GEORGIA-PACIFIC LLC
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE
AREA 2 SOIL AND SEDIMENT
FIELD SAMPLING PLAN

FLOODPLAIN SAMPLING
STRATA



FIGURE
3-a

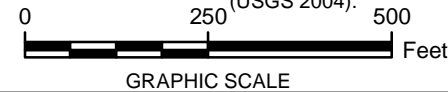


LEGEND:

	EMERGENT WETLAND		CATTAILS/WILLOW SHRUB		EPA SOIL SAMPLE LOCATION
	FLOODPLAIN FOREST		CATTAILS		EXISTING SOIL PCB SAMPLE LOCATION
	LOW TERRACE		FLOODPLAIN FOREST		MAXIMUM PCB RESULTS (MG/KG):
	PREVIOUS CHANNEL		OPEN WATER		1 - 5
	UPLAND AREA		PHRAGMITES		5 - 10
	700 FT CONTOUR		PURPLE LOOSE STRIFE		10 - 50
	WETLAND MEADOW COMPLEX		REED CANARY GRASS		> 50
	FLOODPLAIN FOREST - ASPEN		UPLAND FOREST		EXTENT OF CURRENT IMPOUNDMENT
			WILLOW SHRUB		EPA SEDIMENT SAMPLE LOCATION
					SRI/FS STUDY AREA
					(CH2M HILL 2003)

NOTES:

1. EXTENT OF CURRENT IMPOUNDMENT WAS REPORTED TO BE LOCATED AT USGS TRANSECT AT G10 IN WATER RESOURCE INVESTIGATIONS REPORT 03-4218 (USGS 2004).
2. AERIAL IMAGE DERIVED FROM ORTHOGRAPHIC DATA BY AXIS GEOSPATIAL, LLC. OTSEGO CITY AREA FLOWN SPRING 2010.
3. DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN WINTER 2010/2011.
4. SAMPLE SLO55 EXCLUDED AS IT FALLS WITHIN THE BOUNDARY OF THE CLEAN UP AREA AT OU-4.



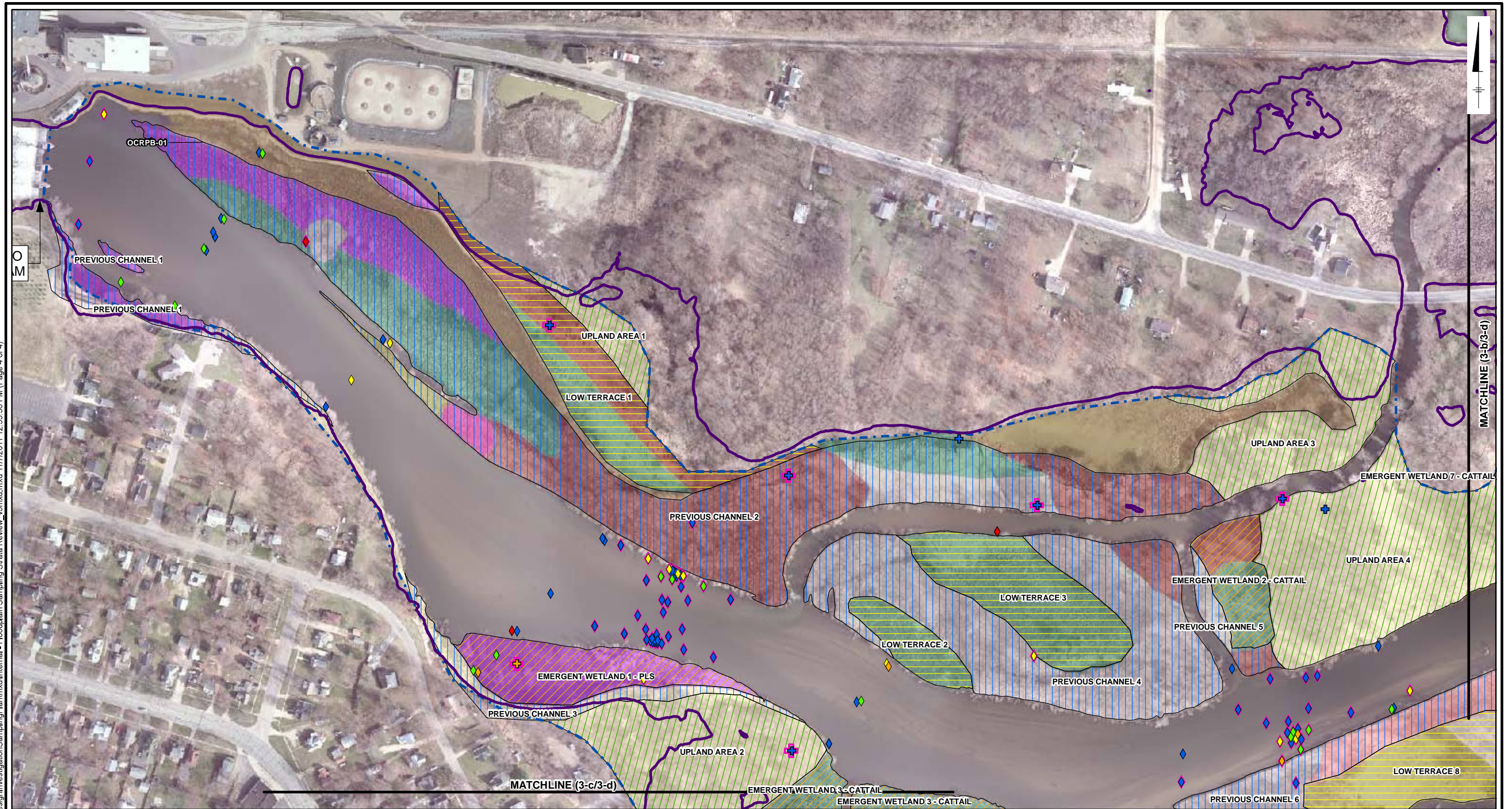
GEORGIA-PACIFIC LLC
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE
AREA 2 SOIL AND SEDIMENT
FIELD SAMPLING PLAN

FLOODPLAIN SAMPLING
STRATA



FIGURE
3-c

City: SYR Div/Group: 90 Created By: mikohberger Last Saved By: spullugurtha
KRS3 - Otsego City (B0064529.0002.00500)
Q:\KRS3\PlanWellDamToOtsegoCityDamPreDesignInvestigationSamplingPlan.mxd Internal - Floodplain Sampling Strata Review_v3.mxd.mxd 11/7/2011 12:55:56 PM (Page 4 of 4)



LEGEND:

EMERGENT WETLAND	CATTAILS/WILLOW SHRUB EXISTING	MAXIMUM PCB RESULTS (MG/KG):	EPA SOIL SAMPLE LOCATION
FLOODPLAIN FOREST	CATTAILS	1 - 5	EXISTING SOIL PCB SAMPLE LOCATION
LOW TERRACE	FLOODPLAIN FOREST	5 - 10	1 - 5
PREVIOUS CHANNEL	OPEN WATER	10 - 50	5 - 10
UPLAND AREA	PHRAGMITES	> 50	10 - 50
700 FT CONTOUR	PURPLE LOOSE STRIFE	EXTENT OF CURRENT IMPOUNDMENT (APPROXIMATE - SEE NOTE 1)	> 50
WETLAND MEADOW COMPLEX	REED CANARY GRASS	EPA SEDIMENT SAMPLE LOCATION	SRI/FS STUDY AREA (CH2M HILL 2003)
FLOODPLAIN FOREST - ASPEN	UPLAND FOREST		
	WILLOW SHRUB		

NOTES:

1. EXTENT OF CURRENT IMPOUNDMENT WAS REPORTED TO BE LOCATED AT USGS TRANSECT AT G10 IN WATER RESOURCE INVESTIGATIONS REPORT 03-4218 (USGS 2004).
2. AERIAL IMAGE DERIVED FROM ORTHOGRAPHIC DATA BY AXIS GEOSPATIAL, LLC. OTSEGO CITY AREA FLOWN SPRING 2010.
3. DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN WINTER 2010/2011.
4. SAMPLE SLO55 EXCLUDED AS IT FALLS WITHIN THE BOUNDARY OF THE CLEAN UP AREA AT OU-4.

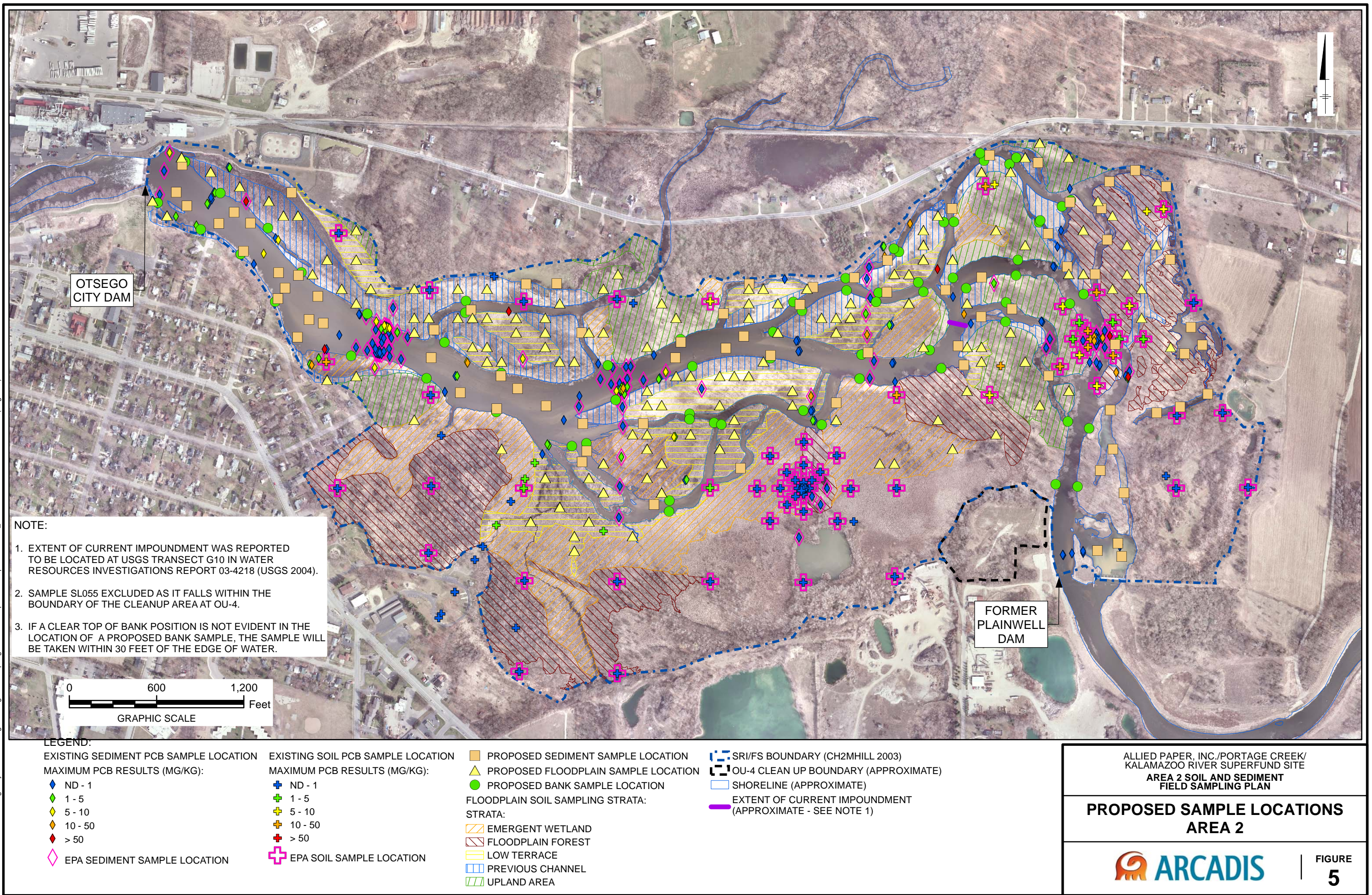


GEORGIA-PACIFIC LLC
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE
AREA 2 SOIL AND SEDIMENT
FIELD SAMPLING PLAN

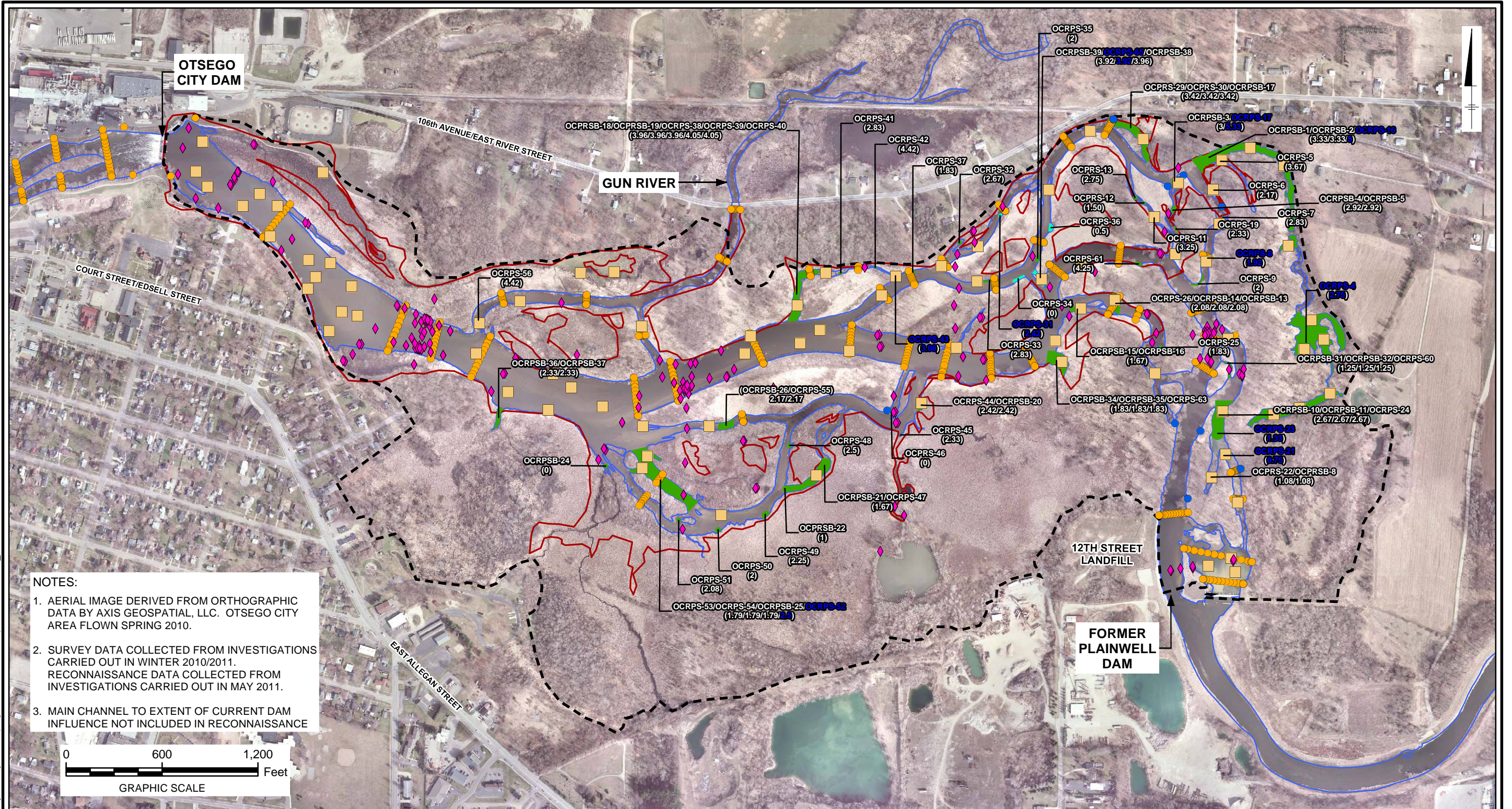
**FLOODPLAIN SAMPLING
STRATA**



FIGURE
3-d



City: SYR Div/Group: 90 Created By: Sruti Pulugurtha Last Saved By: spulugurtha
KRSR - Area 2 Field Sampling Plan (B0064529.0002.00500)
Q:\KRSR\PlanWellDamToOtsegoCityDam\PreDesignInvestigation\SamplingPlan.mxd\Sediment Deposit Areas - Area 2_v4.mxd 11/14/2011 4:22:59 PM



NOTES:

1. AERIAL IMAGE DERIVED FROM ORTHOGRAPHIC DATA BY AXIS GEOSPATIAL, LLC. OTSEGO CITY AREA FLOWN SPRING 2010.
2. SURVEY DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN WINTER 2010/2011. RECONNAISSANCE DATA COLLECTED FROM INVESTIGATIONS CARRIED OUT IN MAY 2011.
3. MAIN CHANNEL TO EXTENT OF CURRENT DAM INFLUENCE NOT INCLUDED IN RECONNAISSANCE

LEGEND:

GEOMORPHIC CLASSIFICATIONS:

- PREVIOUS MAIN CHANNEL
- PREVIOUS CHANNEL
- LOWER TERRACE
- MEDIUM TERRACE
- UPPER TERRACE
- ADDITIONAL RECONNAISSANCE AREA

SEDIMENT DEPOSITION AREA IDENTIFIED DURING AREA RECONNAISSANCE

- COARSE
- FINE
- EXISTING SEDIMENT SAMPLE LOCATIONS THAT FALL WITHIN SHORELINE
- PROPOSED SEDIMENT SAMPLE LOCATIONS
- RIVER TRANSECT
- USGS RIVER TRANSECT

SRI/FS BOUNDARY (CH2MHILL 2003)

- FORMER OTSEGO CITY IMPOUNDMENT BOUNDARY
- CURRENT SHORELINE (APPROXIMATE)

OCRPS-35 – DEPOSITIONAL AREA (BLUE TEXT INDICATES GRAY SILT OR CLAY FOUND IN SAMPLE)
(2) (DEPTH)

ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE
AREA 2 SOIL AND SEDIMENT
FIELD SAMPLING PLAN

PROPOSED SEDIMENT
SAMPLE POINTS



FIGURE
6

Attachment A

Data from Phase 1
Reconnaissance

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPB-01	5/10/2011	0820	12766138.80	353900.43	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-02	5/10/2011	0832	12766177.30	353804.27	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-03	5/10/2011	0900	12766401.18	353664.30	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-04	5/10/2011	0910	12766488.02	353631.11	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-05	5/10/2011	0915	12766426.26	353598.47	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-06	5/10/2011	0950	12766835.00	353197.15	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-07	5/10/2011	1020	12767110.89	353324.9182	Geomorphic boundary location	CDM GPS Unit
OCRPB-08	5/10/2011	1040	12767060.83	353079.77	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-09	5/10/2011	1100	12767417.98	352985.69	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-10	5/10/2011	1204	12768078.69	353036.04	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-11	5/10/2011	1213	12768343.01	353035.56	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-12	5/10/2011	1242	12768766.78	353013.91	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-13	5/10/2011	1300	12769105.06	353129.21	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-14	5/10/2011	1305	12769121.65	353151.36	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-15	5/10/2011	1310	12769100.70	353241.37	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-16	5/10/2011	1445	12767899.52	352613.20	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-17	5/10/2011	1455	12767952.71	352685.28	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-18	5/10/2011	1510	12768015.94	352786.94	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-19	5/10/2011	1545	12768234.36	352654.24	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-20	5/11/2011	0755	12769003.40	352740.95	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-21	5/11/2011	0805	12768799.17	352690.46	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-22	5/11/2011	0815	12768799.89	352801.25	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-23	5/11/2011	0823	12768914.49	352781.74	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-24	5/11/2011	1000	12769965.24	353038.74	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-25	5/11/2011	1006	12769807.46	352964.46	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-26	5/11/2011	1028	12769828.86	353024.71	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-27	5/11/2011	1033	12770176.36	353052.18	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-28	5/11/2011	1034	12770201.25	353009.50	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-29	5/11/2011	1100	12771003.93	352959.01	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-30	5/11/2011	1114	12771179.82	352839.10	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-31	5/11/2011	1125	12770984.32	352612.50	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-32	5/11/2011	1135	12770821.95	352773.68	Geomorphic boundary location	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPB-33	5/11/2011	1200	12770549.58	352755.77	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-34	5/11/2011	1206	12770757.03	352856.72	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-35	5/11/2011	1425	12770863.68	353206.96	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-36	5/11/2011	1445	12770921.31	353098.14	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-37	5/11/2011	1502	12770979.79	353083.13	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-38	5/11/2011	1520	12771082.10	353158.06	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-39	5/11/2011	1525	12771096.23	353243.68	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-40	5/11/2011	1528	12770994.33	353254.99	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-41	5/11/2011	1543	12771044.27	353499.30	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-42	5/11/2011	1640	12771338.76	353372.10	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-43	5/11/2011	1642	12771438.55	353390.98	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-44	5/11/2011	1644	12771546.47	353382.40	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-45	5/11/2011	1655	12771452.71	353447.89	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-46	5/11/2011	1706	12771448.62	353755.23	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-47	5/11/2011	1727	12771388.54	353764.60	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-48	5/11/2011	1740	12771683.57	353747.33	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-49	5/11/2011	1750	12771632.87	353275.31	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-50	5/12/2011	0835	12771813.85	353855.10	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-51	5/12/2011	0845	12771764.86	353936.22	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-52	5/12/2011	0900	12771918.95	353818.53	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-53	5/12/2011	0906	12771894.02	353905.21	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-54	5/12/2011	1045	12772127.31	353583.14	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-55	5/12/2011	1145	12771625.49	353127.99	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-56	5/12/2011	1517	12772715.22	353484.24	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-57	5/12/2011	1715	12772483.37	352623.32	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-58	5/12/2011	1750	12772764.57	352355.94	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-59	5/12/2011	1800	12772748.19	352474.13	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-60	5/13/2011	0952	12770311.45	352396.55	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-61	5/13/2011	1005	12770026.74	352369.98	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-62	5/13/2011	1010	12770199.94	352495.97	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-63	5/13/2011	1015	12769892.16	352383.65	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-64	5/13/2011	1028	12769871.56	352481.3015	Geomorphic boundary location	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPB-65	5/13/2011	1040	12769528.32	352418.34	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-66	5/13/2011	1050	12769623.88	352257.83	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-67	5/13/2011	1052	12769517.71	352264.31	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-68	5/13/2011	1120	12769013.13	352197.2181	Geomorphic boundary location	CDM GPS Unit
OCRPB-69	5/16/2011	0955	12771795.65	352101.05	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-70	5/16/2011	1009	12771456.31	352347.89	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-71	5/16/2011	1022	12771449.29	351995.23	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-72	5/16/2011	1042	12770975.45	351273.50	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-73	5/16/2011	1111	12770962.84	352135.39	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-74	5/16/2011	1127	12770932.60	352311.60	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-75	5/16/2011	1143	12770569.89	352335.13	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-76	5/16/2011	1415	12769141.93	352040.34	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-77	5/16/2011	1431	12769424.47	352022.35	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-78	5/16/2011	1504	12769383.26	351833.74	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-79	5/16/2011	1509	12769302.60	351871.59	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-80	5/16/2011	1608	12769305.33	351099.78	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-81	5/16/2011	1637	12769736.93	351379.3619	Geomorphic boundary location	CDM GPS Unit
OCRPB-82	5/16/2011	1443	12769772.94	351118.41	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-83	5/16/2011	1711	12769200.45	350428.4032	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-84	5/16/2011	1734	12768792.83	350878.49	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-85	5/16/2011	1740	12768799.44	351111.54	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-86	5/16/2011	1825	12768550.76	351498.40	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-87	5/16/2011	1901	12768091.91	352076.54	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-88	5/17/2011	0838	12768634.62	350554.17	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-89	5/17/2011	0905	12768207.65	351163.77	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-90	5/17/2011	1210	12770769.08	351359.14	Geomorphic boundary location	ARCADIS GPS Units
OCRPB-91	5/17/2011	1239	12770126.23	351234.45	Geomorphic boundary location	ARCADIS GPS Units
OCRPF-01	5/9/2011	1504	12767885.24	352208.23	Floodplain soil location	ARCADIS GPS Units
OCRPF-02	5/9/2011	1532	12767723.03	352301.32	Floodplain soil location	ARCADIS GPS Units
OCRPF-03	5/9/2011	1625	12767776.92	352276.267	Floodplain soil location	CDM GPS Unit
OCRPF-04	5/9/2011	1640	12769077.05	352639.66	Floodplain soil location	ARCADIS GPS Units
OCRPF-05	5/9/2011	1700	12768786.53	352000.84	Floodplain soil location	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPF-06	5/10/2011	0810	12766121.63	353850.28	Floodplain soil location	ARCADIS GPS Units
OCRPF-07	5/10/2011	0820	12766138.98	353918.79	Floodplain soil location	ARCADIS GPS Units
OCRPF-08	5/10/2011	0835	12766186.42	353788.13	Floodplain soil location	ARCADIS GPS Units
OCRPF-09	5/10/2011	0900	12766458.23	353646.44	Floodplain soil location	ARCADIS GPS Units
OCRPF-10	5/10/2011	0940	12766760.44	353232.59	Floodplain soil location	ARCADIS GPS Units
OCRPF-11	5/10/2011	0945	12766766.01	353198.97	Floodplain soil location	ARCADIS GPS Units
OCRPF-12	5/10/2011	1000	12766942.48	353198.60	Floodplain soil location	ARCADIS GPS Units
OCRPF-13	5/10/2011	1012	12767035.14	353256.29	Floodplain soil location	ARCADIS GPS Units
OCRPF-14	5/10/2011	1024	12767149.24	353395.26	Floodplain soil location	ARCADIS GPS Units
OCRPF-15	5/10/2011	1045	12767160.14	353020.74	Floodplain soil location	ARCADIS GPS Units
OCRPF-16	5/10/2011	1055	12767396.49	352877.15	Floodplain soil location	ARCADIS GPS Units
OCRPF-17	5/10/2011	1155	12768069.11	352993.94	Floodplain soil location	ARCADIS GPS Units
OCRPF-18	5/10/2011	1205	12768085.29	353085.28	Floodplain soil location	ARCADIS GPS Units
OCRPF-19	5/10/2011	1220	12768613.55	352956.30	Floodplain soil location	ARCADIS GPS Units
OCRPF-20	5/10/2011	1232	12768598.04	353036.77	Floodplain soil location	ARCADIS GPS Units
OCRPF-21	5/10/2011	1245	12768913.29	353037.71	Floodplain soil location	ARCADIS GPS Units
OCRPF-22	5/10/2011	1300	12769105.06	353129.21	Floodplain soil location	ARCADIS GPS Units
OCRPF-23	5/10/2011	1440	12767893.54	352609.35	Floodplain soil location	ARCADIS GPS Units
OCRPF-24	5/10/2011	1450	12767936.32	352635.47	Floodplain soil location	ARCADIS GPS Units
OCRPF-25	5/10/2011	1500	12767939.68	352753.24	Floodplain soil location	ARCADIS GPS Units
OCRPF-26	5/10/2011	1515	12768052.22	352815.96	Floodplain soil location	ARCADIS GPS Units
OCRPF-27	5/10/2011	1530	12768341.37	352829.23	Floodplain soil location	ARCADIS GPS Units
OCRPF-28	5/10/2011	1540	12768262.92	352764.00	Floodplain soil location	ARCADIS GPS Units
OCRPF-29	5/10/2011	1550	12768187.90	352559.01	Floodplain soil location	ARCADIS GPS Units
OCRPF-30	5/10/2011	1640	12768550.98	352701.62	Floodplain soil location	CDM GPS Unit
OCRPF-31	5/10/2011	1650	12768574.38	352817.88	Floodplain soil location	CDM GPS Unit
OCRPF-32	5/10/2011	1710	12768455.77	352647.86	Floodplain soil location	ARCADIS GPS Units
OCRPF-33	5/10/2011	1715	12768491.37	352475.70	Floodplain soil location	ARCADIS GPS Units
OCRPF-34	5/11/2011	0750	12768915.71	352545.00	Floodplain soil location	ARCADIS GPS Units
OCRPF-35	5/11/2011	0800	12768896.97	352581.87	Floodplain soil location	ARCADIS GPS Units
OCRPF-36	5/11/2011	0807	12768847.97	352728.87	Floodplain soil location	ARCADIS GPS Units
OCRPF-37	5/11/2011	0820	12768865.16	352874.76	Floodplain soil location	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPF-38	5/11/2011	0845	12769269.65	352686.30	Floodplain soil location	ARCADIS GPS Units
OCRPF-39	5/11/2011	0857	12769203.86	352968.54	Floodplain soil location	ARCADIS GPS Units
OCRPF-40	5/11/2011	0915	12769575.34	353019.86	Floodplain soil location	ARCADIS GPS Units
OCRPF-41	5/11/2011	0945	12770055.82	352913.17	Floodplain soil location	ARCADIS GPS Units
OCRPF-42	5/11/2011	1007	12769762.57	352957.48	Floodplain soil location	ARCADIS GPS Units
OCRPF-43	5/11/2011	1014	12769762.13	353010.89	Floodplain soil location	ARCADIS GPS Units
OCRPF-44	5/11/2011	1020	12769862.44	353032.70	Floodplain soil location	ARCADIS GPS Units
OCRPF-45	5/11/2011	1105	12771039.66	352941.11	Floodplain soil location	ARCADIS GPS Units
OCRPF-46	5/11/2011	1116	12771167.71	352740.96	Floodplain soil location	ARCADIS GPS Units
OCRPF-47	5/11/2011	1152	12770481.69	352913.61	Floodplain soil location	ARCADIS GPS Units
OCRPF-48	5/11/2011	1210	12770594.39	352855.48	Floodplain soil location	ARCADIS GPS Units
OCRPF-49	5/11/2011	1420	12770851.13	353208.73	Floodplain soil location	ARCADIS GPS Units
OCRPF-50	5/11/2011	1435	12770918.43	353143.68	Floodplain soil location	ARCADIS GPS Units
OCRPF-51	5/11/2011	1450	12770926.30	353072.41	Floodplain soil location	ARCADIS GPS Units
OCRPF-52	5/11/2011	1457	12770988.41	353099.40	Floodplain soil location	ARCADIS GPS Units
OCRPF-53	5/11/2011	1510	12771073.04	353234.14	Floodplain soil location	ARCADIS GPS Units
OCRPF-54	5/11/2011	1530	12770953.90	353270.58	Floodplain soil location	ARCADIS GPS Units
OCRPF-55	5/11/2011	1537	12770992.84	353423.48	Floodplain soil location	ARCADIS GPS Units
OCRPF-56	5/11/2011	1558	12770741.65	353232.31	Floodplain soil location	CDM GPS Unit
OCRPF-57	5/11/2011	1628	12771248.64	353174.60	Floodplain soil location	ARCADIS GPS Units
OCRPF-58	5/11/2011	1635	12771435.59	353290.98	Floodplain soil location	ARCADIS GPS Units
OCRPF-59	5/11/2011	1645	12771470.43	353498.35	Floodplain soil location	ARCADIS GPS Units
OCRPF-60	5/11/2011	1700	12771442.89	353428.18	Floodplain soil location	ARCADIS GPS Units
OCRPF-61	5/11/2011	1710	12771447.40	353849.48	Floodplain soil location	ARCADIS GPS Units
OCRPF-62	5/11/2011	1735	12771667.74	353797.80	Floodplain soil location	ARCADIS GPS Units
OCRPF-63	5/11/2011	1743	12771661.28	353612.43	Floodplain soil location	ARCADIS GPS Units
OCRPF-64	5/12/2011	0820	12771741.97	353915.68	Floodplain soil location	ARCADIS GPS Units
OCRPF-65	5/12/2011	0830	12771810.23	353849.50	Floodplain soil location	ARCADIS GPS Units
OCRPF-66	5/12/2011	0842	12771822.31	353868.51	Floodplain soil location	ARCADIS GPS Units
OCRPF-67	5/12/2011	0856	12771901.37	353832.32	Floodplain soil location	ARCADIS GPS Units
OCRPF-68	5/12/2011	0910	12771973.92	353913.70	Floodplain soil location	ARCADIS GPS Units
OCRPF-69	5/12/2011	0915	12772031.28	353803.88	Floodplain soil location	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPF-70	5/12/2011	0923	12771908.39	353606.91	Floodplain soil location	ARCADIS GPS Units
OCRPF-71	5/12/2011	0935	12771979.05	353508.40	Floodplain soil location	ARCADIS GPS Units
OCRPF-72	5/12/2011	1012	12772068.30	353140.29	Floodplain soil location	ARCADIS GPS Units
OCRPF-73	5/12/2011	1025	12772184.72	353088.07	Floodplain soil location	ARCADIS GPS Units
OCRPF-74	5/12/2011	1037	12772158.09	353484.26	Floodplain soil location	ARCADIS GPS Units
OCRPF-75	5/12/2011	1047	12772114.40	353645.98	Floodplain soil location	ARCADIS GPS Units
OCRPF-76	5/12/2011	1125	12772096.61	352590.55	Floodplain soil location	ARCADIS GPS Units
OCRPF-77	5/12/2011	1138	12771839.17	352980.87	Floodplain soil location	ARCADIS GPS Units
OCRPF-78	5/12/2011	1152	12771548.14	353012.43	Floodplain soil location	ARCADIS GPS Units
OCRPF-79	5/12/2011	1455	12772473.65	353451.42	Floodplain soil location	ARCADIS GPS Units
OCRPF-80	5/12/2011	1500	12772571.79	353434.23	Floodplain soil location	ARCADIS GPS Units
OCRPF-81	5/12/2011	1512	12772732.90	353473.47	Floodplain soil location	ARCADIS GPS Units
OCRPF-82	5/12/2011	1530	12772573.69	353041.58	Floodplain soil location	ARCADIS GPS Units
OCRPF-83	5/12/2011	1542	12772585.57	352798.06	Floodplain soil location	ARCADIS GPS Units
OCRPF-84	5/12/2011	1550	12772744.02	352768.67	Floodplain soil location	ARCADIS GPS Units
OCRPF-85	5/12/2011	1717	12772503.95	352660.39	Floodplain soil location	ARCADIS GPS Units
OCRPF-86	5/12/2011	1745	12772647.85	352280.53	Floodplain soil location	ARCADIS GPS Units
OCRPF-87	5/12/2011	1751	12772795.18	352446.22	Floodplain soil location	ARCADIS GPS Units
OCRPF-88	5/13/2011	0807	12772818.78	351906.44	Floodplain soil location	ARCADIS GPS Units
OCRPF-89	5/13/2011	0812	12773298.11	352056.88	Floodplain soil location	ARCADIS GPS Units
OCRPF-90	5/13/2011	0830	12772639.19	351324.15	Floodplain soil location	ARCADIS GPS Units
OCRPF-91	5/13/2011	0840	12772431.26	351441.38	Floodplain soil location	ARCADIS GPS Units
OCRPF-92	5/13/2011	0853	12772237.22	351675.36	Floodplain soil location	ARCADIS GPS Units
OCRPF-93	5/13/2011	0900	12772200.22	351834.91	Floodplain soil location	ARCADIS GPS Units
OCRPF-94	5/13/2011	0927	12772358.56	352493.12	Floodplain soil location	ARCADIS GPS Units
OCRPF-95	5/13/2011	0955	12770216.55	352352.28	Floodplain soil location	ARCADIS GPS Units
OCRPF-96	5/13/2011	0957	12770241.94	352437.77	Floodplain soil location	ARCADIS GPS Units
OCRPF-97	5/13/2011	1020	12769850.11	352535.95	Floodplain soil location	ARCADIS GPS Units
OCRPF-98	5/13/2011	1031	12769641.13	352342.40	Floodplain soil location	ARCADIS GPS Units
OCRPF-99	5/13/2011	1043	12769565.06	352230.56	Floodplain soil location	ARCADIS GPS Units
OCRPF-100	5/13/2011	1058	12769184.32	352339.17	Floodplain soil location	ARCADIS GPS Units
OCRPF-101	5/13/2011	1102	12769223.88	352278.52	Floodplain soil location	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPF-102	5/13/2011	1116	12769080.58	352232.29	Floodplain soil location	ARCADIS GPS Units
OCRPF-103	5/13/2011	1123	12768899.07	352159.26	Floodplain soil location	ARCADIS GPS Units
OCRPF-104	5/16/2011	0945	12771852.15	352355.88	Floodplain soil location	ARCADIS GPS Units
OCRPF-105	5/16/2011	1012	12771426.43	352322.34	Floodplain soil location	ARCADIS GPS Units
OCRPF-106	5/16/2011	1028	12771287.80	351865.34	Floodplain soil location	ARCADIS GPS Units
OCRPF-107	5/16/2011	1040	12771092.94	351585.08	Floodplain soil location	ARCADIS GPS Units
OCRPF-108	5/16/2011	1106	12770907.50	352039.35	Floodplain soil location	ARCADIS GPS Units
OCRPF-109	5/16/2011	1120	12770991.78	352186.79	Floodplain soil location	ARCADIS GPS Units
OCRPF-110	5/16/2011	1137	12770595.20	352390.01	Floodplain soil location	ARCADIS GPS Units
OCRPF-111	5/16/2011	1150	12770517.75	352117.08	Floodplain soil location	ARCADIS GPS Units
OCRPF-112	5/16/2011	1206	12769915.02	352020.85	Floodplain soil location	ARCADIS GPS Units
OCRPF-113	5/16/2011	1217	12769859.18	351736.57	Floodplain soil location	ARCADIS GPS Units
OCRPF-114	5/16/2011	1407	12768992.88	351838.13	Floodplain soil location	ARCADIS GPS Units
OCRPF-115	5/16/2011	1418	12769168.83	352061.08	Floodplain soil location	ARCADIS GPS Units
OCRPF-116	5/16/2011	1423	12769559.18	352402.15	Floodplain soil location	CDM GPS Unit
OCRPF-117	5/16/2011	1434	12769365.95	351868.03	Floodplain soil location	ARCADIS GPS Units
OCRPF-118	5/16/2011	1443	12769418.73	351769.37	Floodplain soil location	ARCADIS GPS Units
OCRPF-119	5/16/2011	1449	12769480.57	351775.74	Floodplain soil location	ARCADIS GPS Units
OCRPF-120	5/16/2011	1555	12769354.12	351343.56	Floodplain soil location	ARCADIS GPS Units
OCRPF-121	5/16/2011	1627	12769693.48	351449.30	Floodplain soil location	ARCADIS GPS Units
OCRPF-122	5/16/2011	1647	12769778.66	351298.83	Floodplain soil location	ARCADIS GPS Units
OCRPF-123	5/16/2011	1647	12769770.57	350964.34	Floodplain soil location	ARCADIS GPS Units
OCRPF-124	5/16/2011	1716	12769126.37	350599.53	Floodplain soil location	ARCADIS GPS Units
OCRPF-125	5/16/2011	1725	12768958.74	350758.31	Floodplain soil location	ARCADIS GPS Units
OCRPF-126	5/16/2011	1735	12768802.31	351129.26	Floodplain soil location	ARCADIS GPS Units
OCRPF-127	5/16/2011	1745	12768870.54	351243.51	Floodplain soil location	ARCADIS GPS Units
OCRPF-128	5/16/2011	1750	12768961.54	351359.30	Floodplain soil location	ARCADIS GPS Units
OCRPF-129	5/16/2011	1754	12768945.72	351407.04	Floodplain soil location	ARCADIS GPS Units
OCRPF-130	5/16/2011	1813	12768617.67	351615.87	Floodplain soil location	ARCADIS GPS Units
OCRPF-131	5/16/2011	1825	12768526.60	351408.49	Floodplain soil location	ARCADIS GPS Units
OCRPF-132	5/16/2011	1845	12768325.07	351826.59	Floodplain soil location	ARCADIS GPS Units
OCRPF-133	5/16/2011	1858	12768112.10	352119.40	Floodplain soil location	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPF-134	5/16/2011	1900	12768052.04	352014.15	Floodplain soil location	ARCADIS GPS Units
OCRPF-135	5/17/2011	0830	12768482.84	350405.91	Floodplain soil location	ARCADIS GPS Units
OCRPF-136	5/17/2011	0843	12768661.44	350773.53	Floodplain soil location	ARCADIS GPS Units
OCRPF-137	5/17/2011	0858	12768257.15	351271.31	Floodplain soil location	ARCADIS GPS Units
OCRPF-138	5/17/2011	0910	12768170.44	350967.27	Floodplain soil location	ARCADIS GPS Units
OCRPF-139	5/17/2011	0942	12767724.27	351323.79	Floodplain soil location	ARCADIS GPS Units
OCRPF-140	5/17/2011	0951	12767805.35	351485.46	Floodplain soil location	ARCADIS GPS Units
OCRPF-141	5/17/2011	1023	12767594.16	352285.47	Floodplain soil location	ARCADIS GPS Units
OCRPF-142	5/17/2011	1039	12767522.98	352013.51	Floodplain soil location	ARCADIS GPS Units
OCRPF-143	5/17/2011	1053	12767275.34	351598.78	Floodplain soil location	ARCADIS GPS Units
OCRPF-144	5/17/2011	1205	12770752.61	351259.49	Floodplain soil location	ARCADIS GPS Units
OCRPF-145	5/17/2011	1216	12770768.91	351444.98	Floodplain soil location	ARCADIS GPS Units
OCRPF-146	5/17/2011	1222	12770342.84	351505.68	Floodplain soil location	ARCADIS GPS Units
OCRPF-147	5/17/2011	1231	12770104.25	351500.01	Floodplain soil location	ARCADIS GPS Units
OCRPF-148	5/17/2011	1239	12770073.79	350969.90	Floodplain soil location	ARCADIS GPS Units
OCRPF-149	5/13/2011	1150	12767013.57	352589.89	Floodplain soil location	ARCADIS GPS Units
OCRPF-150	5/13/2011	1158	12766816.10	352601.62	Floodplain soil location	ARCADIS GPS Units
OCRPF-151	5/16/2011	0855	12765835.87	353472.55	Floodplain soil location	ARCADIS GPS Units
OCRPF-152	5/16/2011	0900	12765840.14	353506.27	Floodplain soil location	ARCADIS GPS Units
OCRPS-1	5/17/2011	1308	12772795.67	352769.48	Sediment deposition area	ARCADIS GPS Units
OCRPS-2	5/17/2011	1330	12772631.83	352216.19	Sediment deposition area	ARCADIS GPS Units
OCRPS-3	5/17/2011	1351	12772755.46	353273.15	Sediment deposition area	ARCADIS GPS Units
OCRPS-4	5/17/2011	1417	12772448.72	353842.17	Sediment deposition area	ARCADIS GPS Units
OCRPS-5	5/17/2011	1438	12772333.01	353768.18	Sediment deposition area	ARCADIS GPS Units
OCRPS-6	5/17/2011	1459	12772300.44	353589.95	Sediment deposition area	ARCADIS GPS Units
OCRPS-7	5/17/2011	1515	12772353.89	353418.19	Sediment deposition area	ARCADIS GPS Units
OCRPS-8	5/17/2011		12772260.25	353159.55	Sediment deposition area	Approximate
OCRPS-9	5/17/2011		12772205.72	353019.57	Sediment deposition area	Approximate
OCRPS-10	5/17/2011	1619	12771933.65	353388.33	Sediment deposition area	ARCADIS GPS Units
OCRPS-11	5/17/2011	1625	12771920.41	353415.18	Sediment deposition area	ARCADIS GPS Units
OCRPS-12	5/17/2011	1638	12771854.82	353507.70	Sediment deposition area	ARCADIS GPS Units
OCRPS-13	5/17/2011	1646	12771798.90	353541.15	Sediment deposition area	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPS-14	5/17/2011	1657	12771774.90	353531.77	Sediment deposition area	ARCADIS GPS Units (Approx)
OCRPS-15	5/17/2011	1713	12771759.10	353725.84	Sediment deposition area	ARCADIS GPS Units
OCRPS-16	5/18/2011	0811	12772213.47	353737.78	Sediment deposition area	ARCADIS GPS Units
OCRPS-17	5/18/2011	0828	12772045.03	353643.09	Sediment deposition area	ARCADIS GPS Units
OCRPS-18	5/18/2011	0842	12772049.59	353451.01	Sediment deposition area	ARCADIS GPS Units
OCRPS-19	5/18/2011	0911	12772066.89	353355.41	Sediment deposition area	ARCADIS GPS Units
OCRPS-20	5/18/2011	0924	12772080.13	353265.68	Sediment deposition area	ARCADIS GPS Units
OCRPS-21	5/18/2011	1014	12772349.75	351930.46	Sediment deposition area	ARCADIS GPS Units
OCRPS-22	5/18/2011	1025	12772264.16	351776.91	Sediment deposition area	ARCADIS GPS Units
OCRPS-23	5/18/2011	1056	12772313.12	352070.06	Sediment deposition area	ARCADIS GPS Units
OCRPS-24	5/18/2011	1103	12772336.97	352163.35	Sediment deposition area	ARCADIS GPS Units
OCRPS-25	5/18/2011	1230	12771964.91	352604.85	Sediment deposition area	ARCADIS GPS Units
OCRPS-26	5/18/2011	1252	12771676.16	352915.94	Sediment deposition area	ARCADIS GPS Units
OCRPS-27	5/18/2011	1324	12771454.44	352868.73	Sediment deposition area	ARCADIS GPS Units
OCRPS-28	5/18/2011	1332	12771466.89	352838.35	Sediment deposition area	ARCADIS GPS Units
OCRPS-29	5/18/2011	1406	12771864.06	353819.07	Sediment deposition area	ARCADIS GPS Units
OCRPS-30	5/18/2011	1423	12771858.78	353878.32	Sediment deposition area	ARCADIS GPS Units
OCRPS-31	5/18/2011	1456	12770959.47	353427.31	Sediment deposition area	ARCADIS GPS Units
OCRPS-32	5/18/2011	1520	12770700.55	353260.99	Sediment deposition area	Approximate
OCRPS-33	5/18/2011	1543	12770889.16	353034.03	Sediment deposition area	CDM GPS Unit
OCRPS-34	5/18/2011	1607	12771071.06	353041.64	Sediment deposition area	ARCADIS GPS Units (Approx)
OCRPS-35	5/18/2011	1615	12771191.56	353161.93	Sediment deposition area	ARCADIS GPS Units
OCRPS-36	5/18/2011	1631	12771267.25	353355.03	Sediment deposition area	ARCADIS GPS Units
OCRPS-37	5/18/2011	1643	12770409.23	353077.03	Sediment deposition area	ARCADIS GPS Units
OCRPS-38	5/19/2011	0739	12769682.22	352830.60	Sediment deposition area	ARCADIS GPS Units
OCRPS-39	5/19/2011	0807	12769815.11	353071.98	Sediment deposition area	ARCADIS GPS Units
OCRPS-40	5/19/2011	0818	12769703.67	353052.09	Sediment deposition area	ARCADIS GPS Units
OCRPS-41	5/19/2011	0835	12769959.65	353066.24	Sediment deposition area	ARCADIS GPS Units
OCRPS-42	5/19/2011	0844	12770171.49	353096.52	Sediment deposition area	ARCADIS GPS Units
OCRPS-43	5/19/2011	0907	12770300.03	353056.62	Sediment deposition area	ARCADIS GPS Units
OCRPS-44	5/19/2011	0932	12770472.63	352240.71	Sediment deposition area	ARCADIS GPS Units
OCRPS-45	5/19/2011	0956	12770331.79	352057.05	Sediment deposition area	ARCADIS GPS Units

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPS-46	5/19/2011	1006	12770287.16	352185.05	Sediment deposition area	ARCADIS GPS Units
OCRPS-47	5/19/2011	1019	12769871.54	351901.93	Sediment deposition area	ARCADIS GPS Units
OCRPS-48	5/19/2011	1035	12769639.41	351997.40	Sediment deposition area	ARCADIS GPS Units
OCRPS-49	5/19/2011	1134	12769495.03	351556.15	Sediment deposition area	ARCADIS GPS Units
OCRPS-50	5/19/2011	1148	12769192.42	351464.55	Sediment deposition area	ARCADIS GPS Units
OCRPS-51	5/19/2011	1201	12768947.16	351527.53	Sediment deposition area	ARCADIS GPS Units (Approx)
OCRPS-52	5/19/2011	1305	12768662.91	351833.97	Sediment deposition area	ARCADIS GPS Units
OCRPS-53	5/19/2011	1327	12768714.41	351934.41	Sediment deposition area	ARCADIS GPS Units
OCRPS-54	5/19/2011	1338	12769022.65	351652.40	Sediment deposition area	ARCADIS GPS Units (Approx)
OCRPS-55	5/19/2011	1409	12769278.06	352111.52	Sediment deposition area	ARCADIS GPS Units
OCRPS-56	5/19/2011	1434	12767691.18	352776.65	Sediment deposition area	ARCADIS GPS Units
OCRPS-57	5/19/2011	1448	12768101.72	352914.62	Sediment deposition area	ARCADIS GPS Units
OCRPS-58	5/19/2011	1504	12768696.36	352894.24	Sediment deposition area	ARCADIS GPS Units
OCRPS-59	5/19/2011	1540	12768810.92	352523.71	Sediment deposition area	ARCADIS GPS Units
OCRPS-60	5/20/2011	0806	12772390.50	352505.19	Sediment deposition area	ARCADIS GPS Units
OCRPS-61	5/20/2011	0829	12771839.10	353112.08	Sediment deposition area	ARCADIS GPS Units
OCRPS-62	5/20/2011	0848	12771325.59	353241.89	Sediment deposition area	ARCADIS GPS Units (Approx)
OCRPS-63	5/20/2011	0908	12771329.48	352506.23	Sediment deposition area	ARCADIS GPS Units
OCRPS-64	5/20/2011	1020	12767814.42	352167.34	Sediment deposition area	ARCADIS GPS Units
OCRPS-65	5/20/2011	1101	12771218.07	353021.10	Sediment deposition area	ARCADIS GPS Units
OCRPSB-1	5/18/2011	0745	12772405.24	353858.00	Sediment boundary	ARCADIS GPS Units
OCRPSB-2	5/18/2011	0750	12772187.51	353746.02	Sediment boundary	ARCADIS GPS Units
OCRPSB-3	5/18/2011	0820	12772045.00	353643.66	Sediment boundary	ARCADIS GPS Units
OCRPSB-4	5/18/2011	0849	12772053.13	353477.35	Sediment boundary	ARCADIS GPS Units (Approx)
OCRPSB-5	5/18/2011	0854	12772028.17	353447.77	Sediment boundary	ARCADIS GPS Units
OCRPSB-6	5/18/2011	0938	12772020.94	353298.76	Sediment boundary	ARCADIS GPS Units
OCRPSB-7	5/18/2011	0939	12772089.22	353243.44	Sediment boundary	ARCADIS GPS Units
OCRPSB-8	5/18/2011	1031	12772275.47	351810.87	Sediment boundary	ARCADIS GPS Units
OCRPSB-9	5/18/2011	1045	12772312.44	351965.07	Sediment boundary	ARCADIS GPS Units (Approx)
OCRPSB-10	5/18/2011	1138	12772357.47	352265.89	Sediment boundary	ARCADIS GPS Units
OCRPSB-11	5/18/2011	1150	12772388.84	352486.53	Sediment boundary	ARCADIS GPS Units
OCRPSB-12	5/18/2011	1159	12772326.57	352714.84	Sediment boundary	ARCADIS GPS Units (Approx)

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Table A-1 - Reconnaissance Field Notes - Sample Coordinates

Location ID	Date	Time	Easting	Northing	Point classification	Coordinate source
OCRPSB-13	5/18/2011	1302	12771699.11	352901.85	Sediment boundary	ARCADIS GPS Units
OCRPSB-14	5/18/2011	1304	12771661.99	352931.91	Sediment boundary	ARCADIS GPS Units
OCRPSB-15	5/18/2011	1339	12771477.46	352843.50	Sediment boundary	ARCADIS GPS Units
OCRPSB-16	5/18/2011	1347	12771401.51	352841.43	Sediment boundary	ARCADIS GPS Units
OCRPSB-17	5/18/2011	1442	12771622.75	354002.79	Sediment boundary	ARCADIS GPS Units
OCRPSB-18	5/19/2011	0754	12769666.84	352783.22	Sediment boundary	ARCADIS GPS Units
OCRPSB-19	5/19/2011	0757	12769684.60	352850.20	Sediment boundary	ARCADIS GPS Units
OCRPSB-20	5/19/2011	0945	12770443.66	352210.57	Sediment boundary	ARCADIS GPS Units
OCRPSB-21	5/19/2011	1023	12769852.18	351844.75	Sediment boundary	ARCADIS GPS Units
OCRPSB-22	5/19/2011	1033	12769608.98	351725.48	Sediment boundary	ARCADIS GPS Units
OCRPSB-23	5/19/2011	1255	12768521.41	351920.51	Sediment boundary	ARCADIS GPS Units (Approx)
OCRPSB-24	5/19/2011	1259	12768487.20	351867.02	Sediment boundary	ARCADIS GPS Units
OCRPSB-25	5/19/2011	1319	12768715.84	351858.20	Sediment boundary	ARCADIS GPS Units
OCRPSB-26	5/19/2011	1413	12769201.70	352109.21	Sediment boundary	ARCADIS GPS Units
OCRPSB-27	5/19/2011	1419	12768700.89	352034.08	Sediment boundary	ARCADIS GPS Units
OCRPSB-28	5/19/2011	1431	12767696.47	352730.79	Sediment boundary	ARCADIS GPS Units
OCRPSB-29	5/19/2011	1522	12769299.58	353471.16	Sediment boundary	ARCADIS GPS Units
OCRPSB-30	5/20/2011	0754	12772024.56	351415.56	Sediment boundary	ARCADIS GPS Units
OCRPSB-31	5/20/2011	0816	12772391.34	352448.08	Sediment boundary	ARCADIS GPS Units (Approx)
OCRPSB-32	5/20/2011	0819	12772368.25	352507.58	Sediment boundary	ARCADIS GPS Units
OCRPSB-33	5/20/2011	0841	12771485.30	353189.61	Sediment boundary	ARCADIS GPS Units
OCRPSB-34	5/20/2011	0923	12771360.28	352459.24	Sediment boundary	ARCADIS GPS Units
OCRPSB-35	5/20/2011	0926	12771262.29	352563.25	Sediment boundary	ARCADIS GPS Units
OCRPSB-36	5/20/2011	1043	12767788.49	352092.33	Sediment boundary	ARCADIS GPS Units
OCRPSB-37	5/20/2011	1047	12767836.34	352225.25	Sediment boundary	ARCADIS GPS Units
OCRPSB-38	5/20/2011	1118	12771216.24	353010.74	Sediment boundary	ARCADIS GPS Units (Approx)
OCRPSB-39	5/20/2011	1125	12771171.10	353076.35	Sediment boundary	ARCADIS GPS Units

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Table A-2 - Reconnaissance Field Notes - Floodplain Vegetation Assessment

Location ID	Date	Time	Bank Height (ft)	Water Depth (ft)	Water depth BGS (ft)	Ground cover (%)	Ground Cover Species	Tree cover (%)	Tree Cover Species	Shrub cover (%)	Shrub Cover Species
OCRPF-01	5/9/2011	1504		0.58	1	100%	Cattail, Reed canary grass				
OCRPF-02	5/9/2011	1532	1.25		2.33	10%	Sensitive fern, honeysuckle	100%	Red maple, silver maple, red oak	20%	Choke cherry
OCRPF-03	5/9/2011	1625			1.33	100%	Reed canary grass, stinging nettle			80%	Willow
OCRPF-04	5/9/2011	1640	1.08	1		10%	Reed canary grass	100%	Silver maple, Elm		
OCRPF-05	5/9/2011	1700		0.25		100%	Purple loosestrife				
OCRPF-06	5/10/2011	0810		0.75		65%	Purple loosestrife				
OCRPF-07	5/10/2011	0820		0.58		75%	Smartweed, Pickerel weed				
OCRPF-08	5/10/2011	0835		0.33		100%	Cattail				
OCRPF-09	5/10/2011	0900	0.67		0.08	60%	Purple loosestrife	80%	Willow		
OCRPF-10	5/10/2011	0940		0.42		100%	Reed canary grass				
OCRPF-11	5/10/2011	0945	0.83		0.5	100%	Fragmites				
OCRPF-12	5/10/2011	1000		0.08		80%	Purple loosestrife, cattail	5%	Willow	90%	Willow
OCRPF-13	5/10/2011	1012									
OCRPF-14	5/10/2011	1024			1	90%	Reed canary grass	10%	Box elder	25%	Willow
OCRPF-15	5/10/2011	1045	1.08		0.42	60%	Reed canary grass, Skunk cabbage	90%	Willow, Cottonwood		
OCRPF-16	5/10/2011	1055		0.83		80%	Reed canary grass, stinging nettle	60%	Willow	10%	Honeysuckle, Multiflora rose
OCRPF-17	5/10/2011	1155	1.83		1	90%	Reed canary grass, Stinging nettle			80%	Willow
OCRPF-18	5/10/2011	1205			0.33	100%	Cattail, Reed canary grass			10%	Willow
OCRPF-19	5/10/2011	1220	1.58		0.67	90%	Reed canary grass, Garlic mustard	40%	Silver maple, Box elder, Hickory		
OCRPF-20	5/10/2011	1232		0.25		65%	Cattails, Duckweed, Reed canary grass	20%	Willow		
OCRPF-21	5/10/2011	1245	1.5		2	55%	Reed canary grass, golden rod	90%	Silver maple, Ash, Cottonwood	15%	Elderberry, Buckthorn, Raspberry
OCRPF-22	5/10/2011	1300			0.08	60%	Reed canary grass, Skunk cabbage	90%	Silver maple	5%	Spruce, Box elder, Honey suckle
OCRPF-23	5/10/2011	1440			0.58	100%	Reed canary grass, Cattails, Willow	40%	Willow	40%	Willow
OCRPF-24	5/10/2011	1450			0.5	100%	Cattails, Stinging nettle			20%	Willow
OCRPF-25	5/10/2011	1500			0.75	40%	Reed canary grass, Golden rods	100%	Aspen, Cottonwood		Dogwood, Honey suckle, Smartweed, Multiflora rose
OCRPF-26	5/10/2011	1515			0.5	100%	Reed canary grass, Cattails, Stinging nettle			40%	Willow
OCRPF-27	5/10/2011	1530			0.5	75%	Reed canary grass, stinging nettle			100%	Willow
OCRPF-28	5/10/2011	1540				100%	Cattail				
OCRPF-29	5/10/2011	1550		0.67		70%	Reed canary grass, smartweed	30%	Cottonwood, willow	50%	Willow, Buckthorn
OCRPF-30	5/10/2011	1640			0.75	70%	Reed canary grass, smartweed			60%	Willow
OCRPF-31	5/10/2011	1650	2.08		2	90%	Reed canary grass, Garlic mustard	50%	Box elder, Willow	10%	Willow, Honey suckle
OCRPF-32	5/10/2011	1710				100%	Cattail				
OCRPF-33	5/10/2011	1715			1	70%	Reed canary grass, goldenrod	30%	Quaking aspen, Willow	20%	Buckthorn, Green ash, Willow
OCRPF-34	5/11/2011	0750	0.67		0.92	75%	Reed canary grass	50%	Box elder, Willow		
OCRPF-35	5/11/2011	0800			0.75	100%	Reed canary grass, Some cattail			40%	Willow
OCRPF-36	5/11/2011	0807		0.42		100%	Cattail				
OCRPF-37	5/11/2011	0820			1.42	75%	Reed canary grass, Stinging nettle	30%	Silver maple, Box elder	5%	Green ash
OCRPF-38	5/11/2011	0845			0.75	90%	Reed canary grass	20%	Willow	5%	Willow
OCRPF-39	5/11/2011	0857			1.25	80%	Reed canary grass	10%	Silver maple	10%	Willow, Dogwood
OCRPF-40	5/11/2011	0915		0.75		10%	Cattail, Some Stringing nettle			10%	Willow
OCRPF-41	5/11/2011	0945			1.08	100%	Reed canary grass				
OCRPF-42	5/11/2011	1007				100%	Golden rod, grasses			75%	Willow
OCRPF-43	5/11/2011	1014			1.25	20%	Reed canary grass	70%	Willow, Cottonwood		
OCRPF-44	5/11/2011	1020			0.42	90%	Reed canary grass, Purple loosestrife				
OCRPF-45	5/11/2011	1105		0.08		100%	Cattail/Purple loosestrife				
OCRPF-46	5/11/2011	1116		1.83		85%	Reed canary grass, stinging nettle	50%	Box elder, Willow, Green ash		
OCRPF-47	5/11/2011	1152				90%	Reed canary grass	60%	Willow, Box elder	5%	Willow, Box elder
OCRPF-48	5/11/2011	1210			0.17	80%	Reed canary grass, Some purple loosestrife			80%	Willow
OCRPF-49	5/11/2011	1420	2.33		2	100%	Reed canary grass			5%	Willow
OCRPF-50	5/11/2011	1435			2.92	85%	Reed canary grass, Golden rod	90%	Willow, Some box elder	10%	Dogwood
OCRPF-51	5/11/2011	1450			0.92	25%	Willow, Purple loosestrife			80%	Willow
OCRPF-52	5/11/2011	1457		1		85%	Reed canary grass, Purple loosestrife				

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Table A-2 - Reconnaissance Field Notes - Floodplain Vegetation Assessment

Location ID	Date	Time	Bank Height (ft)	Water Depth (ft)	Water depth BGS (ft)	Ground cover (%)	Ground Cover Species	Tree cover (%)	Tree Cover Species	Shrub cover (%)	Shrub Cover Species
OCRPF-53	5/11/2011	1510				80%	Reed canary grass, Stringing nettle	80%	Silver maple	10%	Dogwood
OCRPF-54	5/11/2011	1530			0.92	100%	Reed canary grass, Purple loosestrife				
OCRPF-55	5/11/2011	1537			0.33	100%	Reed canary grass, Purple loosestrife				
OCRPF-56	5/11/2011	1558				60%	Purple loosestrife				
OCRPF-57	5/11/2011	1628			0.83	90%	Reed canary grass				
OCRPF-58	5/11/2011	1635			2	100%	Reed canary grass				
OCRPF-59	5/11/2011	1645		0.83		100%	Cattail				
OCRPF-60	5/11/2011	1700			0.67	100%	Purple loosestrife, Reed canary grass				
OCRPF-61	5/11/2011	1710			2.5	100%	Reed canary grass				
OCRPF-62	5/11/2011	1735			2.5	100%	Reed canary grass				
OCRPF-63	5/11/2011	1743				80%	Reed canary grass	80%	Silver maple		
OCRPF-64	5/12/2011	0820		1.12		15%	Poison ivy	80%	Cottonwood, Willow, Aspen	50%	Green ash, Willow
OCRPF-65	5/12/2011	0830			1.5	45%	Reed canary grass	60%	Cottonwood, Aspen, Sycamore	70%	Green ash
OCRPF-66	5/12/2011	0842			0.75	40%	Purple loosestrife	10%	Willow, Aspen	10%	Dogwood, Willow
OCRPF-67	5/12/2011	0856			0.42	60%	Purple loosestrife				
OCRPF-68	5/12/2011	0910			1.33	100%	Reed canary grass				
OCRPF-69	5/12/2011	0915			1.67	80%	Reed canary grass, Jewel reed	90%	Silver maple		
OCRPF-70	5/12/2011	0923				100%	Reed canary grass				
OCRPF-71	5/12/2011	0935			0.92	100%	Reed canary grass				
OCRPF-72	5/12/2011	1012			1.25	100%	Reed canary grass				
OCRPF-73	5/12/2011	1025			2.5	95%	Reed canary grass	50%	Silver maple	5%	Dogwood
OCRPF-74	5/12/2011	1037			0.83	90%	Reed canary grass, Stringing nettle	40%	Silver maple		
OCRPF-75	5/12/2011	1047			1.25	80%	Reed canary grass				
OCRPF-76	5/12/2011	1125			2.5	85%	Reed canary grass	60%	Sycamore, Ash, Silver maple		
OCRPF-77	5/12/2011	1138			2.75	85%	Reed canary grass, Stringing nettle	50%	Silver maple		
OCRPF-78	5/12/2011	1152			0.83	90%	Reed canary grass	5%	Willow	80%	Willow
OCRPF-79	5/12/2011	1455			1	70%	Reed canary grass	90%	Silver maple	20%	Choke cherry
OCRPF-80	5/12/2011	1500			0	100%	Reed canary grass				
OCRPF-81	5/12/2011	1512			0.75	100%	Reed canary grass				
OCRPF-82	5/12/2011	1530			1.5	75%	Reed canary grass	60%	Silver maple		
OCRPF-83	5/12/2011	1542			0.92	100%	Reed canary grass	50%	Silver maple		
OCRPF-84	5/12/2011	1550			1	75%	Reed canary grass	10%	Silver maple, Sycamore	5%	Honeysuckle, maple
OCRPF-85	5/12/2011	1717			1.5	90%	Reed canary grass	40%	Silver maple, Elm, Sycamore		
OCRPF-86	5/12/2011	1745			2	85%	Reed canary grass	40%	Silver maple, Willow	10%	Honeysuckle
OCRPF-87	5/12/2011	1751			0.67	90%	Lake sedge, Cattail	10%	Willow	20%	Willow, Dogwood
OCRPF-88	5/13/2011	0807			2	60%	Sensitive fern	90%	Sycamore, Ash, Elm	80%	Honeysuckle, Multiflora rose, Dogwood
OCRPF-89	5/13/2011	0812			0	80%	Reed canary grass, Some purple loosestrife	10%	Willow	10%	Honeysuckle
OCRPF-90	5/13/2011	0830				75%	Sensitive fern, reed canary grass, dandelion	90%	Silver maple, Elm	20%	Green ash
OCRPF-91	5/13/2011	0840				100%	Reed canary grass	90%	Silver maple, Elm, Sycamore	40%	Honeysuckle
OCRPF-92	5/13/2011	0853			1.33	70%	Reed canary grass				
OCRPF-93	5/13/2011	0900				90%	Reed canary grass	90%	Silver maple		
OCRPF-94	5/13/2011	0927		1		90%	Reed canary grass	5%	Sycamore, Willow	5%	Willow
OCRPF-95	5/13/2011	0955			0	100%	Cattail			50%	Willow
OCRPF-96	5/13/2011	0957			3.75	85%	Reed canary grass	70%	Box elder, Willow		
OCRPF-97	5/13/2011	1020			1.5	50%	Reed canary grass	80%	Aspen	10%	Willow, Ash
OCRPF-98	5/13/2011	1031			1.5	100%	Reed canary grass	25%	Willow		
OCRPF-99	5/13/2011	1043			1.5	65%	Reed canary grass			65%	Willow
OCRPF-100	5/13/2011	1058			2	90%	Reed canary grass			30%	Young willow
OCRPF-101	5/13/2011	1102			2	100%	Reed canary grass				
OCRPF-102	5/13/2011	1116			2.25	90%	Reed canary grass, Garlic mustard	40%	Willow	20%	Willow
OCRPF-103	5/13/2011	1123			1.5	60%	Reed canary grass, Willow			75%	Willow
OCRPF-104	5/16/2011	0945			1.33	100%	Reed canary grass	40%	Silver maple, Box elder, Green ash		

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Table A-2 - Reconnaissance Field Notes - Floodplain Vegetation Assessment

Location ID	Date	Time	Bank Height (ft)	Water Depth (ft)	Water depth BGS (ft)	Ground cover (%)	Ground Cover Species	Tree cover (%)	Tree Cover Species	Shrub cover (%)	Shrub Cover Species
OCRPF-105	5/16/2011	1012			0.42	100%	Cattail, Reed canary grass	5%	Willow	40%	Willow, Dogwood
OCRPF-106	5/16/2011	1028			0	100%	Cattail				
OCRPF-107	5/16/2011	1040			0	100%	Cattail				
OCRPF-108	5/16/2011	1106			0.5	100%	Cattail				
OCRPF-109	5/16/2011	1120			2	100%	Reed canary grass	15%	Willow (older)	30%	Dogwood
OCRPF-110	5/16/2011	1137			2.33	90%	Reed canary grass	50%	Silver maple, Willow		
OCRPF-111	5/16/2011	1150			0	100%	Cattail				
OCRPF-112	5/16/2011	1206			0	100%	Cattail				
OCRPF-113	5/16/2011	1217			0	100%	Cattail				
OCRPF-114	5/16/2011	1407			0.5	100%	Purple loose strife, Cattail				
OCRPF-115	5/16/2011	1418			0.33	30%	Purple loosestrife, Reed canary grass	60%	Aspen	20%	Aspen
OCRPF-116	5/16/2011	1423			0.58	70%	Reed canary grass	50%	Aspen, Willow	40%	Aspen, Willow
OCRPF-117	5/16/2011	1434		0.33		100%	Cattail				
OCRPF-118	5/16/2011	1443		0.25		80%	Purple loosestrife			10%	Willow
OCRPF-119	5/16/2011	1449			0.5	80%	Reed canary grass			75%	Young Aspen
OCRPF-120	5/16/2011	1555			0	100%	Cattail				
OCRPF-121	5/16/2011	1627			0	100%	Cattail				
OCRPF-122	5/16/2011	1647			0	100%	Cattail			80%	Dogwood
OCRPF-123	5/16/2011	1647			0.42	100%	Skunk cabbage, sedges	40%	Willow, Aspen	60%	Wild raisin, Dogwood
OCRPF-124	5/16/2011	1716				100%	Cattail			20%	Dogwood
OCRPF-125	5/16/2011	1725		0.17		100%	Cattail	40%	Tamarack	10%	Dogwood
OCRPF-126	5/16/2011	1735			0	100%	Cattail				
OCRPF-127	5/16/2011	1745			0	100%	Cattail				
OCRPF-128	5/16/2011	1750		0.33		100%	Cattail			5%	Willow
OCRPF-129	5/16/2011	1754		0.33		40%	Cattail	40%	Willow		
OCRPF-130	5/16/2011	1813		0.33			Money wart, reed canary grass, purple loosestrife	50%	Willow, Aspen	20%	Willow
OCRPF-131	5/16/2011	1825			0.17	100%	Cattail				
OCRPF-132	5/16/2011	1845			0	100%	Cattail				
OCRPF-133	5/16/2011	1858			0.08	80%	Cattail				
OCRPF-134	5/16/2011	1900		0.25		60%	Cattail, Reed canary grass	40%	Silver maple	20%	Dogwood
OCRPF-135	5/17/2011	0830			0.33	90%	Skunk cabbage, sedges	40%	Silver maple	50%	Shrub
OCRPF-136	5/17/2011	0843			0.33	100%	Cattail				
OCRPF-137	5/17/2011	0858			0	100%	Cattail				
OCRPF-138	5/17/2011	0910			0	50%	Skunk cabbage, arrowhead	50%	Silver maple, Ash, Ironwood	25%	Dogwood, Ash
OCRPF-139	5/17/2011	0942			0.29	70%	Reed canary grass	40%	Silver maple, Ash	20%	Dogwood
OCRPF-140	5/17/2011	0951			0.25	70%	Reed canary grass, Jewel reed			80%	Ash, Willow
OCRPF-141	5/17/2011	1023			2.67	25%	Sensitive fern, honeysuckle, garlic mustard	85%	Silver maple, Elm, Black cherry	60%	Honeysuckle
OCRPF-142	5/17/2011	1039		0.25		100%	Cattail				
OCRPF-143	5/17/2011	1053			0.25	90%	Reed canary grass	40%	Silver maple, Willow	20%	Willow
OCRPF-144	5/17/2011	1205			1.67	80%	Equisetum, goldenrod			50%	Willow, Honeysuckle
OCRPF-145	5/17/2011	1216			1.17	100%	Cattails, skunk cabbage				
OCRPF-146	5/17/2011	1222			2	20%	Spice bush, equisetum	10%	Aspen	90%	Honeysuckle, Spice bush
OCRPF-147	5/17/2011	1231			0	100%	Cattail				
OCRPF-148	5/17/2011	1239			0.83	60%	Equisetum, sedges	10%	Willow	50%	Willow, Honeysuckle, Tamarack, Dogwood
OCRPF-149	5/13/2011	1150		0.58		50%	Purple loosestrife	5%	Willow		
OCRPF-150	5/13/2011	1158		0.83		40%	Purple loosestrife				
OCRPF-151	5/16/2011	0855			0.5	80%	Reed canary grass				
OCRPF-152	5/16/2011	0900		0.92		40%	Purple loosestrife				

Notes:

1. BGS - below ground surface

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Table A-3 - Reconnaissance Field Notes - Coring Information

Location ID	Date Collected	Time Collected	Water Depth (ft)	Velocity (ft/s)	Sediment Depth (ft)	Sediment Recovery (ft)	Sediment Penetration (ft)
OCRPS-1	5/17/2011	1308	0.20	0	2.67	2.42	3.58
OCRPS-2	5/17/2011	1330	0.33	0	2.58	2.92	3.67
OCRPS-3	5/17/2011	1351	0.33	0	3.17	1.83	3.17
OCRPS-4	5/17/2011		1.42	Slight	3.83	2.75	3.83
OCRPS-5	5/17/2011	1438	1.33	0	5.17	3.67	5.17
OCRPS-6	5/17/2011	1459	1.83	0.5	3.58	2.17	3.58
OCRPS-7	5/17/2011	1515	1.42	0.4	5.33	2.83	5.33
OCRPS-8	5/17/2011		1.00			1.92	2.42
OCRPS-9	5/17/2011						
OCRPS-10	5/17/2011	1619	1.42	0.6	1.50	1.00	1.50
OCRPS-11	5/17/2011	1625		0.2	3.83	3.25	3.83
OCRPS-12	5/17/2011	1638	0.92	0.3	3.17	1.50	3.17
OCRPS-13	5/17/2011	1646	1.00		5.00	2.75	5.00
OCRPS-14	5/17/2011					3.00	
OCRPS-15	5/17/2011	1713	1.33	0.15	5.50	4.92	5.50
OCRPS-16	5/18/2011	0811	2.17	0.1	5.00	3.33	5.00
OCRPS-17	5/18/2011	0828	2.25	0.1	5.33	3.00	5.33
OCRPS-18	5/18/2011	0842	0.75	0.1	4.50	2.92	4.50
OCRPS-19	5/18/2011	0911	1.67	0.2	3.00	2.33	3.00
OCRPS-20	5/18/2011	0924	0.83	0.3	6.17	3.58	6.17
OCRPS-21	5/18/2011	1014	1.33	0	0.75	0.75	0.75
OCRPS-22	5/18/2011	1025	1.58	0	1.42	1.08	1.42
OCRPS-23	5/18/2011	1056	3.42	0.1	1.67	1.33	1.67
OCRPS-24	5/18/2011	1103	2.00	0	2.75	2.67	2.75
OCRPS-25	5/18/2011	1230	0.83	0	3.33	1.83	3.33
OCRPS-26	5/18/2011	1252	3.08	0	2.83	2.08	2.83
OCRPS-27	5/18/2011	1324	1.67	1	3.58	3.00	3.58
OCRPS-28	5/18/2011	1332	2.08	1	3.67	1.67	3.67
OCRPS-29	5/18/2011	1406	1.25	0.2	5.00	3.08	5.00
OCRPS-30	5/18/2011	1423	1.50	0.3	6.00	3.75	6.00
OCRPS-31	5/18/2011	1456	1.58	1	4.50	2.42	4.50
OCRPS-32	5/18/2011	1520	0.25	0	3.83	2.67	3.83
OCRPS-33	5/18/2011	1543	1.83	0.05	5.67	2.83	5.67
OCRPS-34	5/18/2011	1607	0.50		5.00		5.00
OCRPS-35	5/18/2011	1615	1.50	0.1	3.83	2.00	3.83
OCRPS-36	5/18/2011	1631	1.25	0	4.50		4.50
OCRPS-37	5/18/2011	1643	3.33	0.5	2.25	1.83	2.25
OCRPS-38	5/19/2011	0739	1.67	0	4.75	3.75	4.75
OCRPS-39	5/19/2011	0807	1.25	0	6.00	3.92	6.00
OCRPS-40	5/19/2011	0818	1.33	0	6.00	4.17	6.00
OCRPS-41	5/19/2011	0835	1.00	0	6.40	2.83	6.40
OCRPS-42	5/19/2011	0844	0.50	0	5.83	4.42	5.83
OCRPS-43	5/19/2011	0907	0.67		5.17	3.08	5.17
OCRPS-44	5/19/2011	0932	2.17	0.05	2.42	2.42	2.42
OCRPS-45	5/19/2011	0956	1.50	0	3.17	2.33	3.17

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Table A-3 - Reconnaissance Field Notes - Coring Information

Location ID	Date Collected	Time Collected	Water Depth (ft)	Velocity (ft/s)	Sediment Depth (ft)	Sediment Recovery (ft)	Sediment Penetration (ft)
OCRPS-46	5/19/2011	1006	1.00	0.5	5.00		
OCRPS-47	5/19/2011	1019	1.33	0.1	1.00	1.00	1.00
OCRPS-48	5/19/2011		1.17		4.83	2.50	4.83
OCRPS-49	5/19/2011	1134	1.58	0.05	3.00	2.25	3.00
OCRPS-50	5/19/2011	1148	1.58	0.05	5.25	2.00	5.25
OCRPS-51	5/19/2011	1201	1.17	0	4.83	2.08	4.83
OCRPS-52	5/19/2011	1305	2.33	0	3.17	2.50	3.17
OCRPS-53	5/19/2011	1327	2.67	0.3	3.33	1.83	3.33
OCRPS-54	5/19/2011	1338	1.92	0.3	3.17	1.75	3.17
OCRPS-55	5/19/2011	1409	1.67	1	3.33	2.17	3.33
OCRPS-56	5/19/2011	1434	1.40	0.8	6.00	4.42	6.00
OCRPS-57	5/19/2011	1448	1.83	0.9	5	3.75	5.00
OCRPS-58	5/19/2011	1504	2.33	0.9	4.00	2.83	4.00
OCRPS-59	5/19/2011	1540	1.92	0.2	3.67	3.17	3.67
OCRPS-60	5/20/2011	0806	1.33	0.3	1.42	1.25	1.42
OCRPS-61	5/20/2011	0829	1.25	0	5.58	4.25	5.58
OCRPS-62	5/20/2011	0848	1.08	0.1	3.33	2.42	3.33
OCRPS-63	5/20/2011	0908	1.58	0	1.83	1.83	1.83
OCRPS-64	5/20/2011	1020	2.25	0	3.50	2.33	3.50
OCRPS-65	5/20/2011	1101	0.67	0.2	5.17	3.92	5.17

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Table A-4 - Reconnaissance Field Notes - Core Descriptions

Location ID	Date Processed	Core Top	Core Bottom	Core Units	Core Description	Color
OCRPF-01	5/9/2011	0	12	in	Silty clay	Very dark brown
		12	24	in	Organic peat	Black
		24	36	in	Void space	
OCRPF-02	5/9/2011	0	20	in	Fine to medium sand, trace silt	Very dark grayish brown
		20	34	in	Medium sand	Dark yellowish brown
OCRPF-03	5/9/2011	0	4	in	Clayey silt, organic	Black
		4	16	in	Medium to coarse sand	Dark brown
		16	19	in	Silt	Black
		19	36	in	Medium sand	Black
OCRPF-04	5/9/2011	0	4	in	Clayey silt	Black
		4	24	in	Orange fine sand	Strong brown
		24	36	in	Fine sand, some silt	Very dark brown
OCRPF-05	5/9/2011	0	6	in	Dark brown silt with fine sand	Matrix-Very dark brown, Mottle-Yellowish red
		6	36	in	Silt, trace fine sand	Greenish black
OCRPF-06	5/10/2011	0	7	in	Dark brown gray silt	Very dark brown
		7	36	in	Silty clay	Dark yellowish brown, Subdominant-Black
OCRPF-07	5/10/2011	0	36	in	Medium gray silty clay	Very dark brown
OCRPF-08	5/10/2011	0	3	in	Black organic	
		3	7	in	Silt	
		7	36	in	Gray clay	Matrix-Very dark brown, Mottle-Dark yellowish brown
OCRPF-09	5/10/2011	0	2	in	Black organic detritus	Dark yellowish brown
		2	8	in	Sandy silt	
		8	10	in	Grayish brown sandy silt	Matrix-Very dark brown, Mottle-Dark yellowish brown
		10	32	in	Sandy clay	Matrix-Dark grayish brown, Mottle-Dark yellowish brown
		32	36	in	Clay with trace sand	
OCRPF-10	5/10/2011	0	4	in	Silt, primarily organic	Very dark brown
		4	36	in	Medium sand	Very dark brown
OCRPF-11	5/10/2011	0	8	in	Organic silt	Dark brown, Mix-Very dark brown
		8	10	in	Silt with trace sand	Black
		10	36	in	Medium sand	Matrix-Very dark brown, Mottle-Dark yellowish brown
OCRPF-12	5/10/2011	0	4	in	Organic silt	Very dark brown
		4	10	in	Silt with trace sand	Black
		10	36	in	Clay	Dark grayish brown
OCRPF-13	5/10/2011	0	3	in	Organic silt	Very dark grayish brown
		3	36	in	Clayey silt, trace sand	Black
OCRPF-14	5/10/2011	0	5	in	Organic silt	Black changing to very dark brown
		5	10	in	Clayey sand	Very dark grayish brown
		10	30	in	Sand and gravel	Dark brown
OCRPF-15	5/10/2011	0	5	in	Organic silt	Brown
		5	22	in	Medium sand	Black
		22	28	in	Organic layer with sand	Dark yellowish brown
		28	36	in	Medium sand with organics	Very dark gray
OCRPF-16	5/10/2011	0	4	in	Organic silt	Very dark grayish brown
		4	36	in	Medium sand, trace organic	Very dark gray
OCRPF-17	5/10/2011	0	5	in	Organic silt, trace sand	Dark yellowish brown
		5	15	in	Medium sand	Black
		15	17	in	Organic silt, trace sand	Dark yellowish brown
		17	36	in	Silty sand	Very dark gray
OCRPF-18	5/10/2011	0	9	in	Organic silt	Dark grayish brown, Mix-Black
		9	22	in	Medium sand	Black
		22	26	in	Organic silt, trace sand	Very dark brown
		26	36	in	Silty sand	Very dark gray
OCRPF-19	5/10/2011	0	12	in	Clayey silt	Very dark gray
		12	18	in	Silty sand	Matrix-Very dark gray, Mottle-Dark yellowish brown
		18	26	in	Medium sand	Dark yellowish brown
		26	36	in	Organic silt, trace sand	Dark yellowish brown

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Table A-4 - Reconnaissance Field Notes - Core Descriptions

Location ID	Date Processed	Core Top	Core Bottom	Core Units	Core Description	Color
OCRPF-20	5/11/2011	0	6	in	Organic silt	Very dark gray
		6	12	in	Medium sand, trace silt	Very dark brown
		12	46	in	Organic silt and detritus	Very dark gray
		46	48	in	Silty sand	Very dark gray
OCRPF-21	5/12/2011	0	6	in	Fine sand, trace silt	Very dark brown
		6	12	in	Medium sand, trace silt	Dark yellowish brown
		12	16	in	Silty clay	Black
		16	36	in	Medium sand, trace silt	Dark yellowish brown
OCRPF-22	5/10/2011	0	12	in	Organic silt	Very dark gray
		12	36	in	Medium sand and gravel	Very dark grayish brown
OCRPF-23	5/10/2011	0	1	in	Organic silt	Black
		1	10	in	Medium sand	Very dark grayish brown
		10	14	in	Medium sand, trace silt	Very dark grayish brown
		14	36	in	Silty clay	Very dark brown
OCRPF-24	5/10/2011	0	36	in	Silt	Very dark grayish brown
OCRPF-25	5/10/2011	0	1	in	Organic	
		1	36	in	Coarse sand and gravel	Dark yellowish brown
OCRPF-26	5/10/2011	0	4	in	Sandy silt	Very dark gray
		4	10	in	Medium sand	Dark yellowish brown
		10	36	in	Silty sand	Very dark grayish brown
OCRPF-27	5/10/2011	0	5	in	Silty clay, trace sand	Very dark gray
		5	10	in	Orange sand	Dark yellowish brown
		10	15	in	Silty clay	Dark grayish brown, Mix-Very dark brown, Mottle-Dark yellowish brown
		15	32	in	Gray sandy silt	Very dark brown
		32	36	in	Silty clay	Very dark grayish brown, Mix-Black
OCRPF-28	5/10/2011	0	36	in	Gray brown clayey silt	Very dark grayish brown
OCRPF-29	5/10/2011	0	3	in	Organic silt	Black
		3	36	in	Sand and gravel	Dark yellowish brown
OCRPF-30	5/10/2011	0	4	in	Sandy silt with organics	Black
		4	36	in	Medium sand with organics	Dark yellowish brown
OCRPF-31	5/10/2011	0	5	in	Sandy silt with organics	Black
		5	36	in	Medium sand with organics	Dark yellowish brown
OCRPF-32	5/10/2011	0	6	in	Silty clay	Dark grayish brown, Mottle-Dark yellowish brown
		6	10	in	Silty clay with organic detritus	Dark grayish brown, Mottle-Dark yellowish brown
		10	32	in	Clayey silt	Black
		32	36	in	Gray medium sand	Dark gray
OCRPF-33	5/10/2011	0	36	in	Medium sand with organics	Dark yellowish brown
OCRPF-34	5/11/2011	0	1	in	Sandy silt with organics	Black
		1	36	in	Medium sand	Dark yellowish brown
OCRPF-35	5/11/2011	0	6	in	Sandy silt with organics	Black
		6	22	in	Medium sand	Dark yellowish brown
		22	24	in	Black sandy silt	Very dark gray
		24	36	in	Medium sand with organics	Dark yellowish brown
OCRPF-36	5/11/2011	0	3	in	Sandy silt with organics	Black
		3	36	in	Silty sand	Very dark grayish brown, Mottle-Dark yellowish brown
OCRPF-37	5/11/2011	0	5	in	Sandy silt	Black
		5	36	in	Medium sand with organic	Dark yellowish brown
OCRPF-38	5/11/2011	0	9	in	Sandy silt	Dark brown
		9	36	in	Silty sand	Very dark grayish brown
OCRPF-39	5/11/2011	0	6	in	Sandy silt	Black
		6	34	in	Medium sand with organic streaking	Dark yellowish brown
		34	36	in	Medium sand	Very dark grayish brown
OCRPF-40	5/11/2011	0	20	in	Sandy silt	Very dark grayish brown
		20	36	in	Medium sand with organic silt	Dark yellowish brown
OCRPF-41	5/11/2011	0	1	in	Sandy silt	Black
		1	36	in	Sand, gravel, silt	Very dark brown
OCRPF-42	5/11/2011	0	2	in	Sandy silt with organic	Black
		2	36	in	Coarse sand	Very dark grayish brown

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Location ID	Date Processed	Core Top	Core Bottom	Core Units	Core Description	Color
OCRPF-43	5/11/2011	0	2	in	Sandy silt	Black
		2	36	in	Sand and gravel	Very dark grayish brown
OCRPF-44	5/11/2011	0	4	in	Sandy silt	Black and very dark grayish brown
		4	36	in	Medium sand, sand and gravel	Very dark grayish brown
		36	40	in	Sandy silt	Very dark grayish brown
OCRPF-45	5/11/2011	0	36	in	Gray sandy silt	Very dark grayish brown
OCRPF-46	5/11/2011	0	36	in	Clayey silt	Black
OCRPF-47	5/11/2011	0	4	in	Sandy silt	Black
		4	36	in	Medium sand with organic streaking	Dark yellowish brown
OCRPF-48	5/11/2011	0	2	in	Organic silt	Black
		2	30	in	Sand and gravel	Very dark grayish brown
		30	36	in	Gray sandy silt	Very dark gray
OCRPF-49	5/11/2011	0	2	in	Detritus organic	Black
		2	36	in	Coarse sand, gravel	Very dark grayish brown
OCRPF-50	5/11/2011	0	1	in	Organic detritus and medium sand	Black
		1	30	in	Medium sand, gravel	Very dark grayish brown
		30	36	in	Gray sandy silt	
OCRPF-51	5/11/2011	0	5	in	Sandy silt	Very dark gray
		5	14	in	Gray clayey silt	
		14	20	in	Sandy silt and shells	Very dark gray
		20	36	in	Dark gray silt	
OCRPF-52	5/11/2011	0	11	in	Coarse sand	Very dark grayish brown
		11	24	in	Light gray sandy silt, trace sand	
		24	36	in	Dark gray sandy silt	Greenish black
OCRPF-53	5/11/2011	0	5	in	Clay	Very dark gray
		5	12	in	Gray clay	
		12	36	in	Gray sandy silt	
OCRPF-54	5/11/2011	0	2	in	Black silt with organics	Black
		2	36	in	Sand and gravel	Very dark grayish brown
OCRPF-55	5/11/2011	0	5	in	Silt with organics	Very dark grayish brown
		5	9	in	Silt, sand and gravel	Very dark gray
		9	15	in	Dark gray silt, trace sand	
		15	36	in	Dark gray sandy silt	
OCRPF-56	5/11/2011	0	4	in	Organic silt	Very dark gray
		4	36	in	Sand and gravel	Very dark grayish brown
OCRPF-57	5/11/2011	0	9	in	Silty clay with organics	Very dark grayish brown
		9	11	in	Silty sand	Very dark grayish brown
		11	36	in	Gray silty clay, some sand	
OCRPF-58	5/11/2011	0	5	in	Organic silt	Black
		5	32	in	Medium sand	Dark yellowish brown
		32	36	in	Gray sandy silt	
OCRPF-59	5/11/2011	0	36	in	Gray silt and clay, trace sand, some organics/detritus	
OCRPF-60	5/11/2011	0	36	in	Silt and clay, trace gray sand	
OCRPF-61	5/11/2011	0	5	in	Silty clay with organic detritus	Black
		5	36	in	Medium sand	Dark yellowish brown
OCRPF-62	5/11/2011				Similar condition to F-61 (CDM core)	
OCRPF-63	5/11/2011	0	36	in	Silty clay	Very dark grayish brown
OCRPF-64	5/12/2011	0	3	in	Clayey silt with organics	Very dark gray
		3	36	in	Light brown medium to coarse sand	Very dark grayish brown
OCRPF-65	5/12/2011	0	2	in	Clayey silt with organics	Black
		2	5	in	Medium sand	Very dark grayish brown
		5	36	in	Coarse sand	Very dark grayish brown
OCRPF-66	5/12/2011	0	3	in	Clayey silt with organics	Very dark grayish brown
		3	5	in	Gray sandy silt	
		5	30	in	Coarse sand	Very dark grayish brown
		30	31	in	Black coarse sand	
		31	36	in	Coarse sand	Very dark grayish brown
OCRPF-67	5/12/2011	0	36	in	Grayish brown clayey silt	Very dark grayish brown
OCRPF-68	5/12/2011	0	36	in	Grayish brown clayey silt	Very dark grayish brown

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OCRPF-69	5/12/2011	0	36	in	Grayish brown clayey silt	Very dark grayish brown
OCRPF-70	5/12/2011	0	2	in	Organic and silt	Black
		2	24	in	Sand and gravel	Very dark grayish brown
OCRPF-71	5/12/2011	0	5	in	Sandy silt with organics	Black
		5	20	in	Coarse sand and gravel	Very dark grayish brown
		20	36	in	Sandy silt	Very dark grayish brown
OCRPF-72	5/12/2011	0	3	in	Organic silt	Black
		3	20	in	Coarse sand and gravel	Very dark grayish brown
		20	36	in	Medium sand with shell fragments	Very dark grayish brown
OCRPF-73	5/12/2011	0	30	in	Grayish silty clay	Very dark grayish brown
		30	31	in	Medium sand lens	Dark yellowish brown
		31	36	in	Grayish silty clay	Very dark grayish brown
OCRPF-74	5/12/2011	0	4	in	Sandy silt with organics	Very dark grayish brown
		4	10	in	Medium sand	Very dark brown
		10	36	in	Coarse sand and gravel	Very dark brown
OCRPF-75	5/12/2011	0	10	in	Sandy silt with organics	Black
		10	32	in	Silty fine to medium sand	Brown
		32	36	in	Gray medium sand	
OCRPF-76	5/12/2011	0	18	in	Brownish gray silty clay, trace fine sand	Very dark grayish brown
		18	36	in	Silty fine sand	Very dark grayish brown
OCRPF-77	5/12/2011	0	8	in	Silty clay	Very dark grayish brown
		8	18	in	Gray silty clay	
		18	36	in	Fine to medium sand	Very dark grayish brown
OCRPF-78	5/12/2011	0	4	in	Silty clay	Very dark gray
		4	32	in	Coarse sand with shell fragments	Dark yellowish brown
		32	36	in	Gray sandy silt	
OCRPF-79	5/12/2011	0	26	in	Silty clay with organics	Very dark grayish brown
		26	28	in	Medium sand	Dark grayish brown
		28	36	in	Sandy silt	Dark grayish brown
OCRPF-80	5/12/2011	0	4	in	Detritus and silt	Black
		4	28	in	Gray silt with organics, trace sand	
		28	36	in	Dark gray silty fine sand	
OCRPF-81	5/12/2011	0	24	in	Silty clay with organics	Very dark grayish brown
		24	36	in	Gray sandy silt	
OCRPF-82	5/12/2011	0	30	in	Silt and clay with trace sand	Very dark grayish brown
		30	36	in	Gray silty clay	
OCRPF-83	5/12/2011	0	4	in	Organic silt	Black
		4	24	in	Coarse sand and gravel	Dark grayish brown
OCRPF-84	5/12/2011	0	6	in	Organic silt	Black
		6	21	in	Coarse sand	Very dark grayish brown
		21	36	in	Medium silty sand	Very dark gray
OCRPF-85	5/12/2011	0	10	in	Sandy silt	Black
		10	36	in	Medium sand and gravel	Dark grayish brown
OCRPF-86	5/12/2011	0	10	in	Organic silt	Black
		10	35	in	Dark gray sandy clay	
		35	36	in	Coarse sand	Dark yellowish brown
OCRPF-87	5/12/2011	0	36	in	Organic silt and detritus	Black
OCRPF-88	5/13/2011	0	8	in	Sandy silt	Very dark grayish brown
		8	12	in	Fine sand	Very dark grayish brown
		12	36	in	Medium sand	Dark yellowish brown
OCRPF-89	5/13/2011	0	4	in	Organic detritus, silt with trace sand	Very dark grayish brown
		4	18	in	Gray medium sand	
		18	36	in	Gray medium sand, some gravel	
OCRPF-90	5/13/2011	0	30	in	Fine sand with organic detritus	Dark yellowish brown
OCRPF-91	5/13/2011	0	3	in	Sandy silt	Very dark grayish brown
		3	12	in	Fine sand with organics and some gravel	Very dark grayish brown
OCRPF-92	5/13/2011	0	3	in	Sandy silt	Black
		3	8	in	Coarse sand and gravel	Dark grayish brown
		8	14	in	Gray medium sand with shell fragments	
		14	36	in	Gray sandy silt	

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OCRPF-93	5/13/2011	0	8	in	Sandy clay	Very dark grayish brown
		8	18	in	Medium sand	Dark yellowish brown
		18	28	in	Sandy clay	Very dark grayish brown
		28	30	in	Detritus layer	Very dark grayish brown
		30	36	in	Sandy clay	Very dark grayish brown
OCRPF-94	5/13/2011	0	8	in	Organic detritus and shell fragments	Very dark gray
		8	11	in	Medium sand with organic streaking	Dark grayish brown
		11	24	in	Silty medium sand, trace gravel	Very dark grayish brown
		24	36	in	Gray sandy silt	
OCRPF-95	5/13/2011	0	36	in	Clayey silt, trace sand and organic detritus	Black
OCRPF-96	5/13/2011	0	3	in	Sandy silt with organics	Black
		3	36	in	Coarse sand and gravel	Dark grayish brown
OCRPF-97	5/13/2011	0	2	in	Organic silt	Black
		2	12	in	Coarse sand	Dark grayish brown
		12	36	in	Coarse sand and gravel	Dark grayish brown
OCRPF-98	5/13/2011	0	3	in	Sandy clay with organics	Black
		3	36	in	Coarse sand	Very dark grayish brown
OCRPF-99	5/13/2011	0	2	in	Silty clay with organics	Black
		2	34	in	Medium sand	Very dark grayish brown
		34	36	in	Dark gray black silty medium sand	
OCRPF-100	5/13/2011	0	3	in	Sandy clay with organics	Black
		3	36	in	Coarse sand	Very dark grayish brown
OCRPF-101	5/13/2011	0	3	in	Sandy clay with organics	Black
		3	36	in	Coarse sand	Very dark grayish brown
OCRPF-102	5/13/2011	0	3	in	Sandy clay with organics	Black
		3	36	in	Coarse sand	Very dark grayish brown
		3	36	in	Sandy clay with organics	Black
OCRPF-103	5/13/2011	0	3	in	Sandy clay with organics	Black
		3	36	in	Coarse sand	Very dark grayish brown
		3	36	in	Coarse sand	Very dark grayish brown
OCRPF-104	5/16/2011	0	10	in	Silty clay	Very dark grayish brown
		10	12	in	Medium sand with organic streaking	Dark grayish brown
		12	36	in	Silty sand	Very dark grayish brown
OCRPF-105	5/16/2011	0	30	in	Grayish clayey silt with organic detritus	
		30	36	in	Medium sand	Dark grayish brown
OCRPF-106	5/16/2011	0	10	in	Organic detritus	
		10	24	in	Dark gray silt with organics	Very dark gray
		24	30	in	Dark gray silt, trace sand	
		30	36	in	Sandy silt	Very dark gray
OCRPF-107	5/16/2011	0	4	in	Organic detritus	
		4	10	in	Gray silty clay	
		10	36	in	Black organic layer with detritus/peat, sulphuric odor	
OCRPF-108	5/16/2011	0	10	in	Black organic detritus	
		10	30	in	Silty clay with organics	Very dark grayish brown
		30	36	in	Fine sand and gravel	Very dark gray
OCRPF-109	5/16/2011	0	10	in	Organic silt, trace sand	Black
		10	24	in	Medium sand	Very dark grayish brown
		24	36	in	Silty sand	Very dark grayish brown
OCRPF-110	5/16/2011	0	12	in	Clay with organics	Very dark gray
		12	18	in	Gray clay	
		18	36	in	Dark gray clayey silt	
OCRPF-111	5/16/2011	0	7	in	Organic silt	Very dark grayish brown
		7	36	in	Black to gray silt with organics, changing to brown with depth	Very dark grayish brown
OCRPF-112	5/16/2011	0	4	in	Silt with organic detritus	Very dark grayish brown
		4	20	in	Gray clayey silt with organics	
		20	30	in	Gray silty sand	
		30	36	in	Gray sandy silt	
OCRPF-113	5/16/2011	0	6	in	Organic detritus	Very dark grayish brown
		6	24	in	Gray brown silt with organics	
		24	30	in	Black detritus layer	
		30	36	in	Greenish gray clayey medium sand	
OCRPF-114	5/16/2011	0	36	in	Dark gray silt, trace fine sand, little bit of petroleum odor	

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OCRPF-115	5/16/2011	0	2	in	Silty clay with organics	Very dark gray
		2	36	in	Coarse sand with shell fragments	Very dark grayish brown
OCRPF-116	5/16/2011	0	2	in	Silty clay	Very dark gray
		2	36	in	Coarse sand	Very dark grayish brown
OCRPF-117	5/16/2011	0	30	in	Gray silty clay, trace sand	
		30	36	in	Dark gray silt	
OCRPF-118	5/16/2011	0	4	in	Gray sandy silt	
		4	14	in	Silty clay	
		14	36	in	Silty clay, trace sand	
OCRPF-119	5/16/2011	0	2	in	Silty clay	Very dark gray
		2	33	in	Coarse sand	Very dark grayish brown
		33	36	in	Dark gray coarse sand	Very dark gray
OCRPF-120	5/16/2011	0	8	in	Organic detritus	Dark yellowish brown
		8	36	in	Clayey silt	Dark yellowish brown
OCRPF-121	5/16/2011	0	4	in	Light brown and gray clay with organic detritus	
		4	24	in	Light brown and gray clay	
		24	36	in	Black detritus and light gray clay	
OCRPF-122	5/16/2011	0	1	in	Detritus	
		1	3	in	Clay with detritus	Dark yellowish brown
		3	36	in	Gray clay	Dark yellowish brown
OCRPF-123	5/16/2011	0	1	in	Organic silt and detritus	Black
		1	16	in	Gray clay	Dark yellowish brown
		16	24	in	Gray silty fine sand	
		24	36	in	Black peat	
OCRPF-124	5/16/2011	0	2	in	Black organic detritus	
		2	12	in	Brown silty clay	Very dark grayish brown
		12	36	in	Organic peat	
OCRPF-125	5/16/2011	0	2	in	Black organic detritus	
		2	8	in	Clay	Dark grayish brown
		8	36	in	Black peat	
OCRPF-126	5/16/2011	0	24	in	Peat	
		24	36	in	Black organic silt and peat	
OCRPF-127	5/16/2011	0	20	in	Peat	
		20	36	in	Silt with organics	Black
OCRPF-128	5/16/2011	0	4	in	Peat, gray silt with organics	
		4	8	in	Black organic silt	Black
		8	36	in	Peat	
OCRPF-129	5/16/2011	0	3	in	Organic silt and detritus	Black
		3	9	in	Gray silt with organics	
		9	36	in	Black silt with organics	
OCRPF-130	5/16/2011	0	3	in	Organic silt and detritus, gray clayey silt	Black
		3	14	in	Black organic layer	
		14	36	in	Dark gray to black clayey silt	
OCRPF-131	5/16/2011	0	6	in	Organic detritus	Very dark grayish brown
		6	18	in	Organic silt	Black
		18	36	in	Black organic silt and detritus	
OCRPF-132	5/16/2011	0	24	in	Brownish gray organic silt	
		24	36	in	Black silty detritus	
OCRPF-133	5/16/2011	0	3	in	Black detritus	
		3	36	in	Black organic silt	
OCRPF-134	5/16/2011	0	1	in	Black detritus	
		1	20	in	Black organic silt with detritus	
		20	36	in	Brown detritus	Very dark grayish brown
OCRPF-135	5/17/2011	0	10	in	Black detritus	Black
		10	36	in	Brown detritus	Dark grayish brown
OCRPF-136	5/17/2011	0	36	in	Organic peat	Dark grayish brown
OCRPF-137	5/17/2011	0	10	in	Organic peat	Dark grayish brown
		10	24	in	Black organic silt and detritus	
		24	36	in	Brown organic peat	Dark grayish brown

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OCRPF-138	5/17/2011	0	6	in	Black organic peat	
		6	10	in	Black silt with detritus	
		10	16	in	Brown detritus	Very dark grayish brown
		16	20	in	Black silt	
		20	36	in	Brown detritus	
OCRPF-139	5/17/2011	0	3	in	Black detritus	
		3	26	in	Black clayey silt with detritus	
		26	36	in	Brown detritus	Very dark grayish brown
OCRPF-140	5/17/2011	0	12	in	Black silt and detritus	
		12	20	in	Brown detritus	Very dark grayish brown
		20	30	in	Black silt and detritus	
		30	36	in	Brown detritus	
OCRPF-141	5/17/2011	0	12	in	Medium sand, trace detritus	Very dark grayish brown
		12	24	in	Reddish medium sand	Dark reddish brown
		24	36	in	Medium sand	Dark yellowish brown
OCRPF-142	5/17/2011	0	36	in	Black silt and detritus	
OCRPF-143	5/17/2011	0	3	in	Black organic silt with detritus	Black
		3	4	in	Coarse sand	Very dark grayish brown
		4	34	in	Dark gray to brown sandy silt with detritus	
		34	36	in	Black sandy silt	
OCRPF-144	5/17/2011	0	32	in	Clay	Dark yellowish brown
		32	36	in	Natural gray brown clay	
OCRPF-145	5/17/2011	0	33	in	Clay	Dark yellowish brown
		33	36	in	Clay, becoming more gray	
OCRPF-146	5/17/2011	0	22	in	Clay	Dark yellowish brown
		22	32	in	Natural gray and brown clay	
		32	36	in	Brown peat	
OCRPF-147	5/17/2011	0	20	in	Clay	Dark yellowish brown
		20	30	in	Gray and black clay with detritus	
		30	36	in	Gray clay with detritus	
OCRPF-148	5/17/2011	0	14	in	Clay	Dark yellowish brown
		14	30	in	Olive green gray and light brown clay	
		30	32	in	Gray and black organic clay	
		32	36	in	Gray silty clay	
OCRPF-149	5/13/2011	0	36	in	Light gray silt	
OCRPF-150	5/13/2011	0	36	in	Dark charcoal silt	
OCRPF-151	5/16/2011	0	8	in	Silty clay	Black
		8	10	in	Silty sand	Black
		10	14	in	Sandy silt	Black
		14	24	in	Medium sand	Very dark grayish brown
		24	36	in	Medium sand	Dark yellowish brown
OCRPF-152	5/16/2011	0	1	in	Dark gray silt	
		1	36	in	Light gray silty clay	
OCRPS-1	5/17/2011	0	13	in	Dark brown loose silt, trace fine sand	
		13	16	in	Dark gray fine to medium sand, trace silt	
		16	19	in	Dark brown silt, trace fine sand, trace shells	
		19	27	in	Gray fine sand, trace medium, trace coarse sand, trace shells	
		27	29	in	Dark gray brown fine sand, trace silt	
OCRPS-2	5/17/2011	0	27	in	Brown silt, trace fine sand, slight odor	
		27	35	in	Gray brown fine sand, trace silt	
OCRPS-3	5/17/2011	0	12	in	Dark brown loose silt, trace fine sand, slight odor	
		12	20	in	Dark gray brown silt, little clay, trace fine sand	
		20	22	in	Gray brown fine sand, trace shells	
OCRPS-4	5/17/2011	0	8	in	Dark brown fine sand, trace medium to coarse sand, trace organics (twigs/leaves)	
		8	24	in	Gray brown fine sand, trace medium to coarse sand	
		24	26	in		
		26	28	in	Dark gray brown silt and clay, trace shells, odor	
		28	29	in	Dark gray fine sand, trace shells	
		29	30	in	Gray silt, little fine sand	
		30	33	in	Dark gray brown fine sand, trace silt, slight odor	

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Table A-4 - Reconnaissance Field Notes - Core Descriptions

Location ID	Date Processed	Core Top	Core Bottom	Core Units	Core Description	Color
OCRPS-5	5/17/2011	0	11	in	Gray brown loose silt, trace clay, sheens, odor: Upper 2"-Dark brown fine sand, little silt;	
		11	22	in	Interbedded silt and fine sand, trace shells, trace sheens, odor	
		22	30	in	Dark brown silt, trace clay, trace organics (leaves/wood)	
		30	38	in	Dark brown silt, trace clay, trace organics (leaves/wood)	
		38	44	in	Gray brown fine and medium sand, little coarse sand, trace fine gravel	
OCRPS-6	5/17/2011	0	3	in	Dark gray brown fine sand, trace silt, trace medium to coarse sand	
		3	17	in	Gray brown silt, trace organics, trace fine sand, trace sheens, slight odor	
		17	26	in	Brown and dark gray brown fine sand, little silt, lower portion is gray brown fine sand, trace silt, trace shells	
OCRPS-7	5/17/2011	0	34	in	Interbedded brown and dark brown fine sand, trace medium to coarse sand, trace silt	
OCRPS-8	5/17/2011	0	6	in	Gray brown fine to coarse sand, trace fine to medium gravel	
OCRPS-10	5/17/2011	6	23	in	Gray silty clay, odor	
		0	12	in	Fine and coarse sand, little fine to medium gravel	
OCRPS-11	5/17/2011	0	2	in	Brown silt	
		2	39	in	Interbedded dark gray brown and brown fine sand and fine medium sand, trace coarse sand, trace fine gravel	
OCRPS-12	5/17/2011	0	6	in	Dark brown loose silt, trace organics, trace fine sand	
		6	18	in	Gray brown fine to coarse sand, trace fine to medium gravel	
OCRPS-13	5/17/2011	0	12	in	Dark gray fine sand, trace silt, trace organics	
		12	18	in	Dark gray brown fine sand, little silt, sheens, odor	
		18	29	in	Dark gray brown silty clay, odor, sheens	
		29	33	in	Gray brown fine sand, trace organics (degraded wood)	
OCRPS-14	5/17/2011	0	12	in	Gray brown silty fine to medium sand	
		12	18	in	Gray brown fine to medium sand	
		18	24	in	Medium gray clayey silt, slight odor	
		24	36	in	Light gray fine sand, some silt	
OCRPS-15	5/17/2011	0	59	in	Gray brown fine to medium sand, trace coarse sand, trace silt	
OCRPS-16	5/18/2011	0	6	in	Gray brown silt, little fine sand	
		6	14	in	Gray clay silt, trace fine sand, sheen, slight odor	
		14	36	in	Dark gray brown fine sand, trace silt, trace organics (shell/vegetation), slight sheen	
OCRPS-17	5/18/2011	0	24	in	Dark gray brown fine sand, trace organics (leaves/wood/shells), trace silt	
		24	26	in	Gray silty clay, sheens, odor	
		26	36	in	Dark gray fine sand, little organics (wood/shells), trace silt, sheens	
OCRPS-18	5/18/2011	0	35	in	Interbedded brown and dark gray brown fine sand, trace organics (wood/shells), trace silt	
OCRPS-19	5/18/2011	0	12	in	Dark gray brown fine sand, trace medium to coarse sand, trace organics	
		12	20	in	Medium gray silty clay	
		20	28	in	Dark gray silty fine sand, trace organics (shells)	
OCRPS-20	5/18/2011	0	28	in	Gray brown fine sand, gray fine to medium sand, trace organics (wood/shells)	
		28	34	in	Gray fine to medium sand, little coarse sand, trace shells	
		34	43	in	Medium gray silty clay, odor, sheens	
OCRPS-21	5/18/2011	0	5	in	Dark gray brown silty fine sand	
		5	9	in	Light gray silty clay, slight odor, trace sheens	
OCRPS-22	5/18/2011	0	5	in	Black loose silt, trace organics	
		5	13	in	Gray brown fine to coarse sand, fine to medium gravel	
OCRPS-23	5/18/2011	0	6	in	Dark gray brown silty fine to coarse sand, trace fine gravels, trace organics	
		6	16	in	Gray silty clay, trace fine sand, odor, sheens	
OCRPS-24	5/18/2011	0	19	in	Interbedded gray brown fine sand, dark gray fine to coarse sand, trace fine to medium gravel, trace silt, trace shells	
		19	30	in	Medium gray silty clay, odor, sheens	
		30	32	in	Gray brown fine to medium sand, trace fine to medium gravel, trace silt	
OCRPS-25	5/18/2011	0	13	in	Dark gray brown silt, trace fine sand, trace organics	
		13	17	in	Gray brown fine to medium sand, trace coarse sand, trace sand and gravel	
		17	22	in	Dark gray silty fine sand, trace medium to coarse sand, slight sheen	
OCRPS-26	5/18/2011	0	10	in	Dark gray brown silt, trace shells, trace wood	
		10	14	in	Dark gray brown fine sand, trace silt	
		14	17	in	Gray fine sand, trace silt, trace shells	
		17	25	in	Dark gray brown silt, little organics (wood)	
OCRPS-27	5/18/2011	0	15	in	Brown grading to dark gray fine to medium sand, trace coarse sand, trace shells	
		15	31	in	Medium gray silty clay, trace organics, sheen, odor	
		31	36	in	Brown organic silt	
OCRPS-28	5/18/2011	0	1	in	Gray fine sand, trace silt	
		1	20	in	Medium gray silty clay, sheens, odor	

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Area 2/Otsego City Impoundment Investigation Field Sampling Plan

Table A-4 - Reconnaissance Field Notes - Core Descriptions

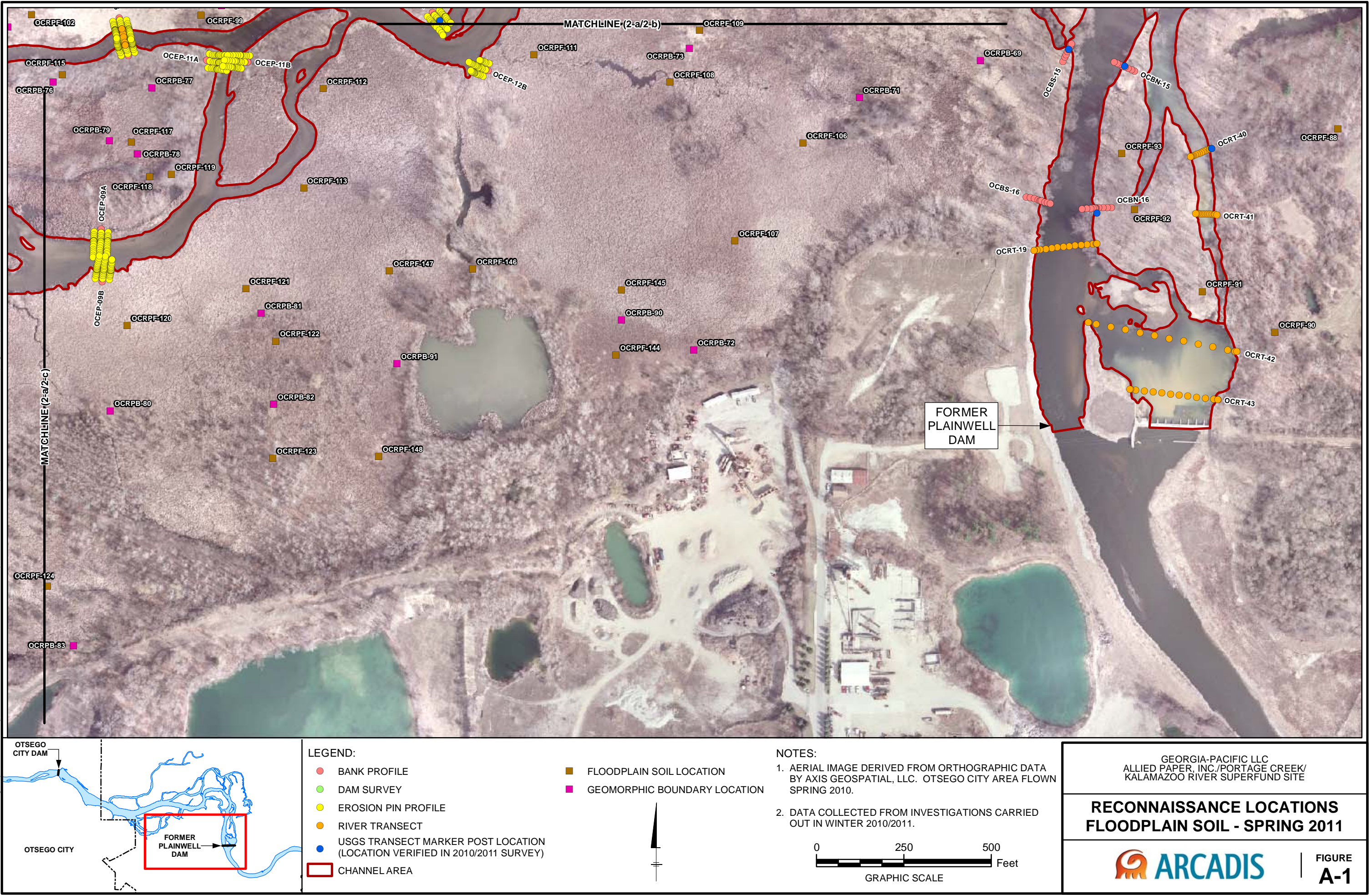
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		24	26	in	Dark gray clayey silt, trace fine sand, trace sheens	
		26	37	in	Dark gray fine sand, trace silt, trace shells	
OCRPS-30	5/18/2011	0	6	in	Brown silty fine sand	
		6	28	in	Dark gray silt, trace fine sand, slight sheen in silt	
		28	45	in	Gray fine to medium sand, trace coarse sand, trace fine gravel, trace shells	
OCRPS-31	5/18/2011	0	5	in	Brown fine to coarse sand, trace fine gravel, trace shells	
		5	25	in	Dark gray silty clay, trace fine sand, slight odor, trace sheen, interbedded sand	
		25	29	in	Light gray calcareous fine sand	
OCRPS-32	5/18/2011	0	7	in	Brown silty fine sand	
		7	24	in	Gray brown grading to dark gray fine to medium sand, little coarse sand, trace shells,	
		24	32	in	Dark gray brown silty clay, stiff	
OCRPS-33	5/18/2011	0	10	in	Dark brown silty fine sand, trace shells	
		10	24	in	Dark brown silty fine sand, little organics, sheens, odor	
		24	30	in	Gray silty clay, trace shells, no odor, no sheen	
		30	34	in	Dark brown organic silt, trace fine sand	
OCRPS-35	5/18/2011	0	6	in	Gray brown silty fine sand	
		6	14	in	Gray silty fine sand, trace organics, sheen; odor; Upper 2/10"-gray silty clay, odor	
		14	24	in	Dark brown silty fine sand, trace organics, sheen	
OCRPS-37	5/18/2011	0	1	in	Gray brown fine sand, trace shells	
		1	18	in	Dark gray silty clay, slight odor, sheens	
		18	22	in	Gray brown silty clay	
OCRPS-38	5/19/2011	0	12	in	Dark brown blue silt, trace fine sand	
		12	17	in	Dark gray brown fine to medium sand, trace coarse sand, trace shells	
		17	30	in	Brown organic silt, trace fine sand, trace shells	
OCRPS-39	5/19/2011	0	22	in	Gray brown fine sand, trace shells	
		22	35	in	Dark gray brown fine to coarse sand	
		35	47	in	Silty clay, slight odor, trace organics	
OCRPS-40	5/19/2011	0	14	in	Dark gray brown silty fine sand	
		14	36	in	Dark gray loose silt, trace organics	
		36	40	in	Gray brown sand, trace silt	
		40	45	in	Dark gray silty clay, moderate odor	
		45	50	in	Light gray brown fine sand, trace shells	
OCRPS-41	5/19/2011	0	14	in	Dark gray silty clay, slight odor	
		14	21	in	Dark gray brown silty fine sand, little organics	
		21	34	in	Dark gray brown fine to coarse sand, trace fine gravel, trace organics (wood/shell)	
OCRPS-42	5/19/2011	0	3	in	Dark gray brown silty clay, slight odor, sheen	
		3	30	in	Brown loose silt, trace fine sand, trace organics	
		30	53	in	Gray brown fine to medium sand, little coarse sand, trace shells	
OCRPS-43	5/19/2011	0	12	in	Dark gray silty clay, slight odor, sheens	
		12	20	in	Brown silty fine sand, trace organics	
		20	25	in	Dark gray clayey silt, trace fine sand, slight odor, sheens	
OCRPS-44	5/19/2011	0	4	in	Dark gray brown silty fine sand	
		4	24	in	Light gray to gray silty clay	
		24	29	in	Brown silty fine sand	
OCRPS-45	5/19/2011	0	9	in	Dark brown organic silt	
		9	18	in	Light gray fine sand	
		18	28	in	Dark brown very loose silt, trace fine sand	
OCRPS-47	5/19/2011	0	5	in	Black loose silt, trace organics	
		5	12	in	Gray silty fine sand	
		12	18	in	Dark brown loose silt	
OCRPS-48	5/19/2011	0	8	in	Olive brown silty clay, trace fine sand, trace fine to medium gravel	
		8	10	in	Dark gray silty clay, slight odor, sheens, trace organics	
		10	19	in	Dark brown highly degraded organics, peat	
		19	30	in	Brown silty clay, trace organics (vegetation)	
					Light gray silty fine sand, trace fine gravel, trace organics, trace shells	

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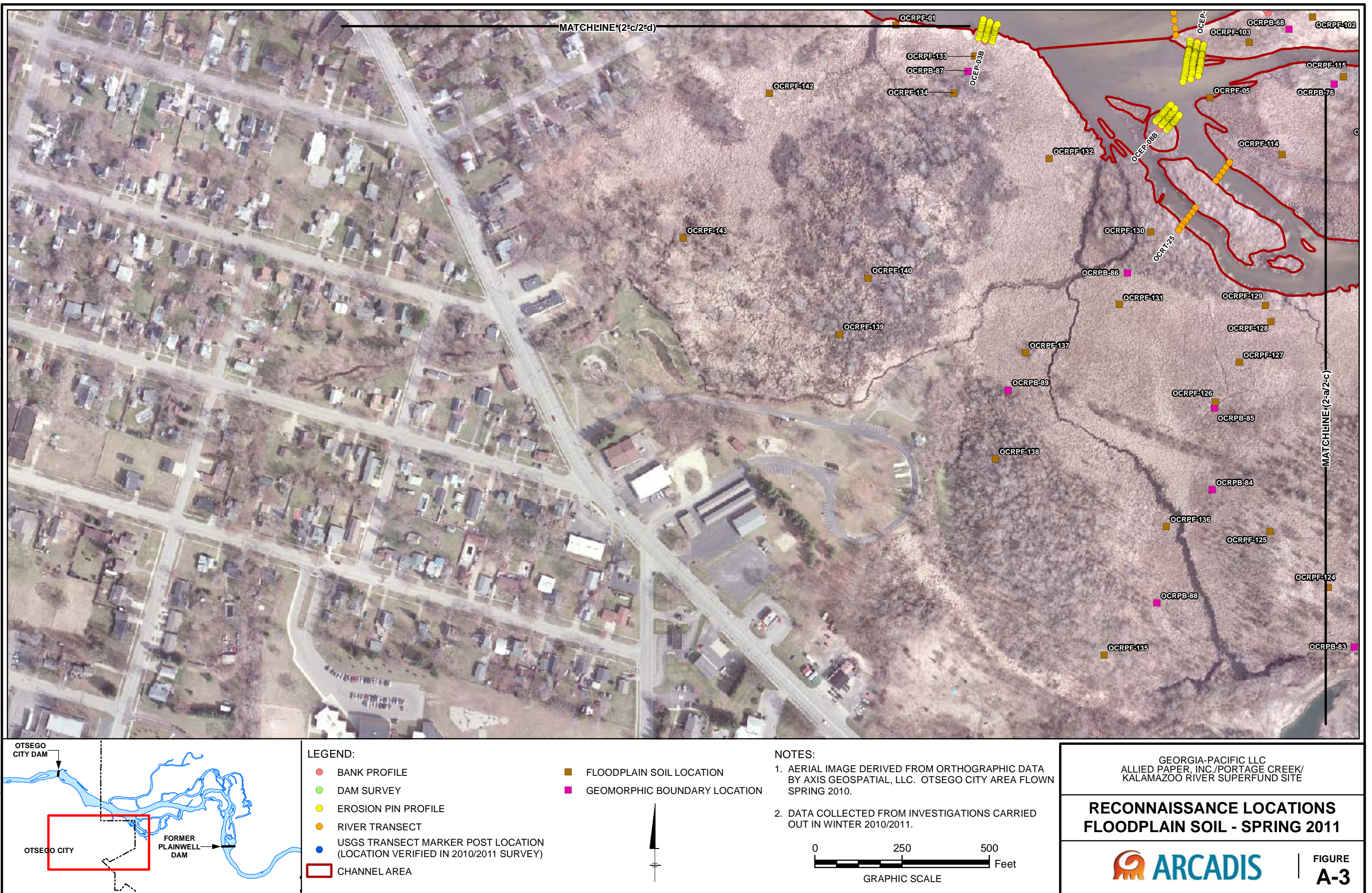
Table A-4 - Reconnaissance Field Notes - Core Descriptions

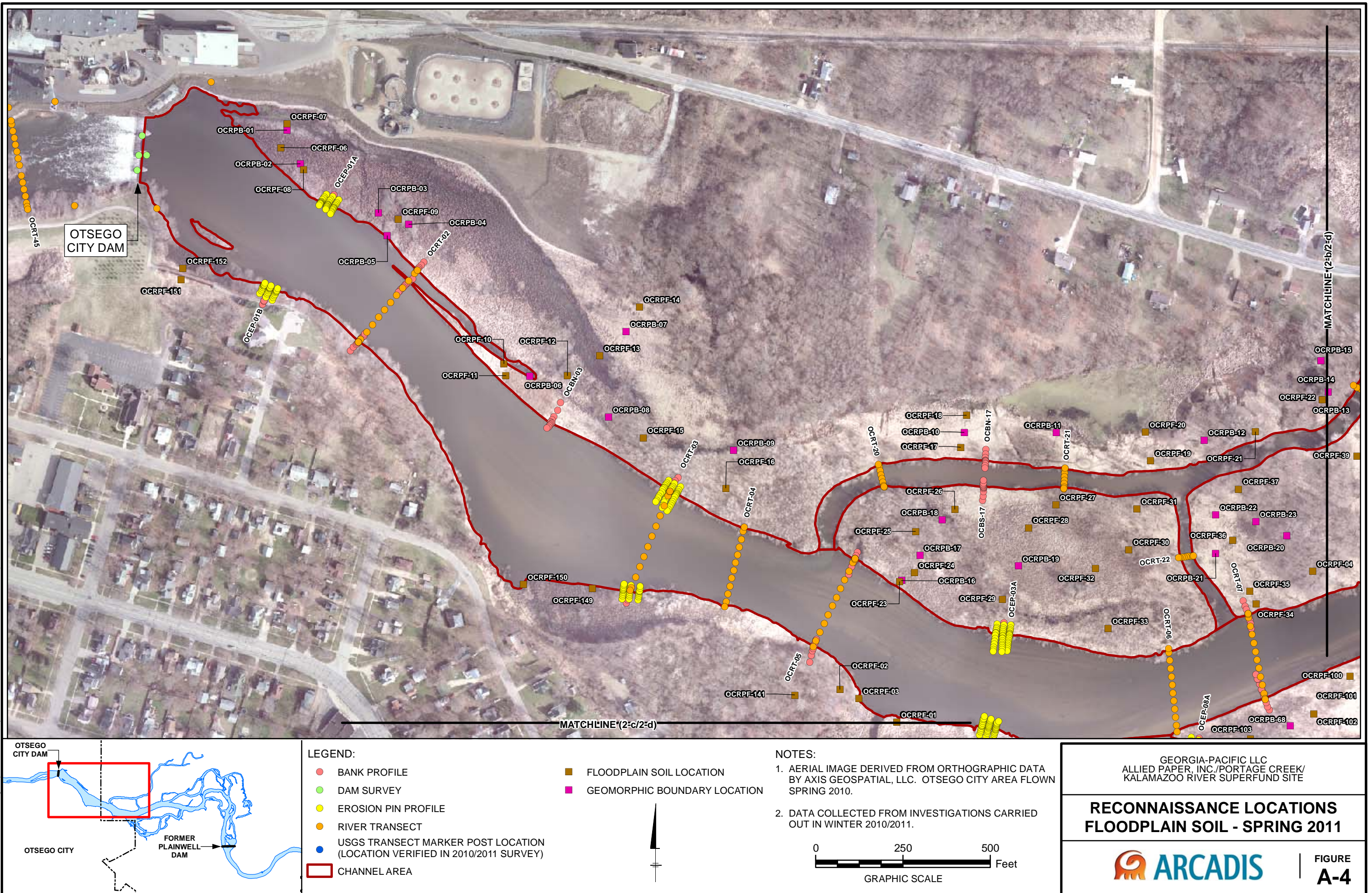
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		9	16	in	Dark brown highly degraded organics, peat	
		16	27	in	Light gray fine sand, trace silt, trace shells	
OCRPS-50	5/19/2011	0	8	in	Dark brown loose silt, trace fine sand	
		8	15	in	Dark brown highly degraded organics, peat	
		15	24	in	Gray fine sand, trace medium to coarse sand, trace silt	
OCRPS-51	5/19/2011	0	12	in	Gray brown loose silt, trace fine sand	
		12	22	in	Dark brown highly degraded organics, peat	
		22	25	in	Gray brown silty fine sand, trace shells	
OCRPS-52	5/19/2011	0	7	in	Dark brown silty fine sand, little organics	
		7	16	in	Peat, dark brown highly degraded organics	
		16	21	in	Gray coarse silty clay, trace fine sand, trace organics	
		21	26	in	Light gray silty fine sand, peat	
		26	30	in	Gray brown silty sand	
OCRPS-53	5/19/2011	0	7	in	Gray brown fine sand, trace silt	
		7	19	in	Dark brown highly degraded organics, peat	
		19	22	in	Brown silty clay, trace fine sand, trace organics (rootlets)	
OCRPS-54	5/19/2011	0	8	in	Gray brown fine sand	
		8	21	in	Dark brown highly degraded organics (peat)	
OCRPS-55	5/19/2011	0	4	in	Gray brown fine sand, trace organics (roots)	
		4	26	in	Brown silty clay, little fine sand	
OCRPS-56	5/19/2011	0	33	in	Orange brown grading to dark gray fine to medium sand, little coarse sand	
		33	53	in	Dark gray brown silty clay, trace organics, odor at the top	
OCRPS-57	5/19/2011	0	21	in	Orange brown grading to gray brown fine to medium sand, trace coarse sand	
		21	45	in	Dark gray silty clay, slight odor	
OCRPS-58	5/19/2011	0	12	in	Orange brown fine to medium grain, fine to medium sand, trace coarse sand	
		12	32	in	Dark brown highly degraded organics, slight odor	
		32	34	in	Gray brown fine to medium sand	
OCRPS-59	5/19/2011	0	17	in	Orange brown grading to gray brown fine to medium sand, trace coarse sand	
		17	32	in	Dark brown organic silt/peat	
		32	38	in	Gray brown fine sand, trace silt	
OCRPS-60	5/20/2011	0	11	in	Brown grading to dark gray brown fine to medium sand, trace coarse sand	
		11	13	in	Dark gray silty clay, odor, sheens	
		13	15	in	Dark gray silty fine sand, trace shells, sheens, odor	
OCRPS-61	5/20/2011	0	29	in	Gray brown fine sand, trace silt, trace shells	
		29	40	in	Dark gray brown fine to medium sand, trace shells	
		40	51	in	Highly degraded organic silt and fine sand, no odor	
OCRPS-62	5/20/2011	0	10	in	Dark brown sandy silt, trace organics	
		10	16	in	Dark brown highly degraded organic silt, trace fine sand	
		16	22	in	Clayey silt, little fine sand	
		22	27	in	Light gray brown clayey silt, little fine sand	
		27	29	in	Light gray to light calcareous sand and gravel	
OCRPS-63	5/20/2011	0	7	in	Dark brown loose silt, trace fine sand, trace organics, trace sheen, trace shells	
		7	14	in	Brown silty fine sand, trace organics	
		14	22	in	Orange gray calcareous fine sand, little silt	
OCRPS-64	5/20/2011	0	11	in	Dark brown mostly degraded organics and silt	
		11	17	in	Dark brown highly degraded organic silt/peat	
		17	28	in	Light orange brown grading to dark gray brown silty fine sand, trace organics (wood)	
OCRPS-65	5/20/2011	0	17	in	Brown gray to dark gray fine sand, trace silt, trace medium to coarse sand, trace fine gravel, trace sheen	
		17	24	in	Dark gray coarse sand, fine gravel, little fine to medium sand	
		24	40	in	Gray clayey silt, trace fine sand	
		40	45	in	Dark gray brown fine sand, trace organics	
		45	47	in	Dark brown sandy organic silt	

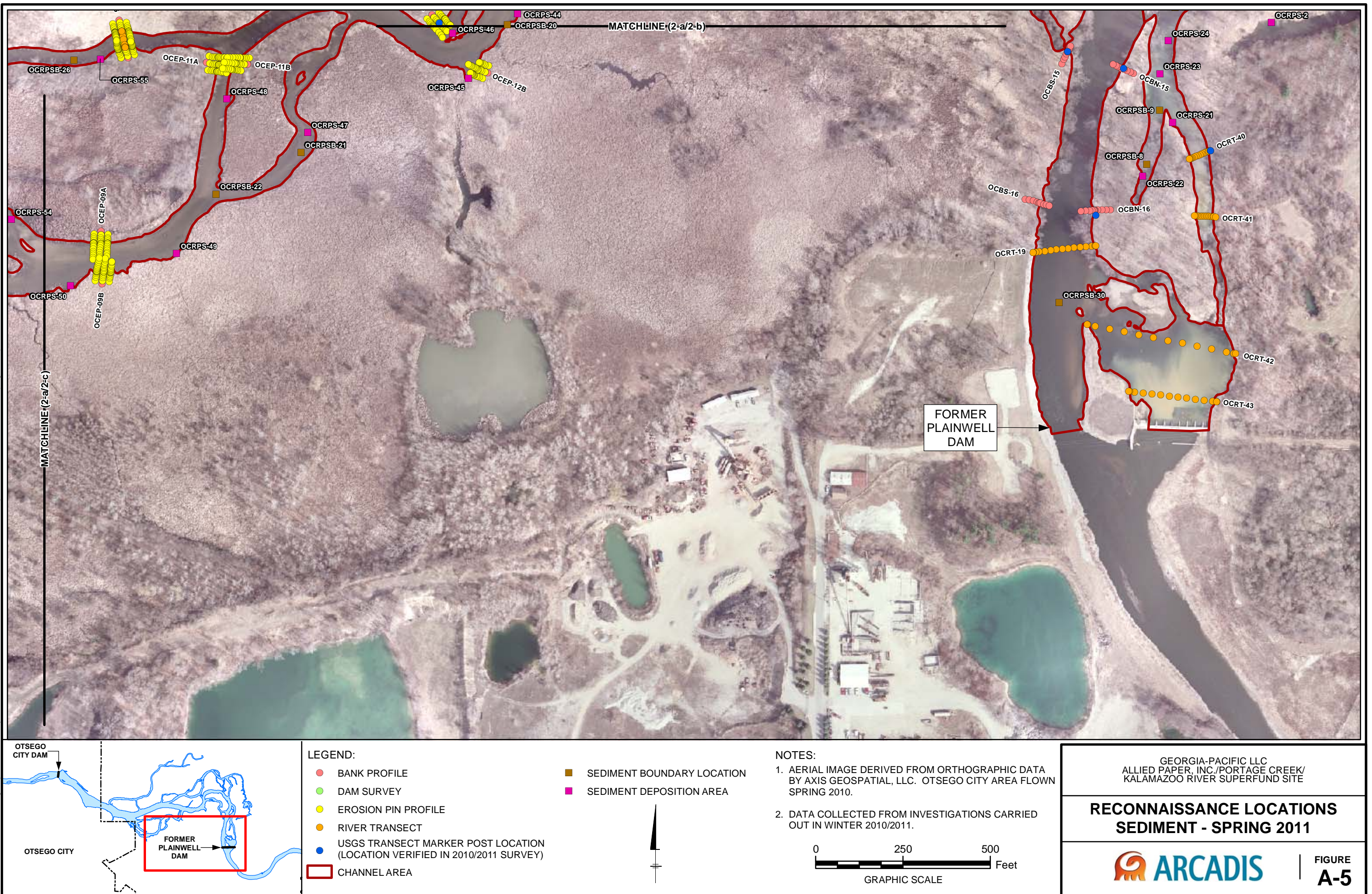
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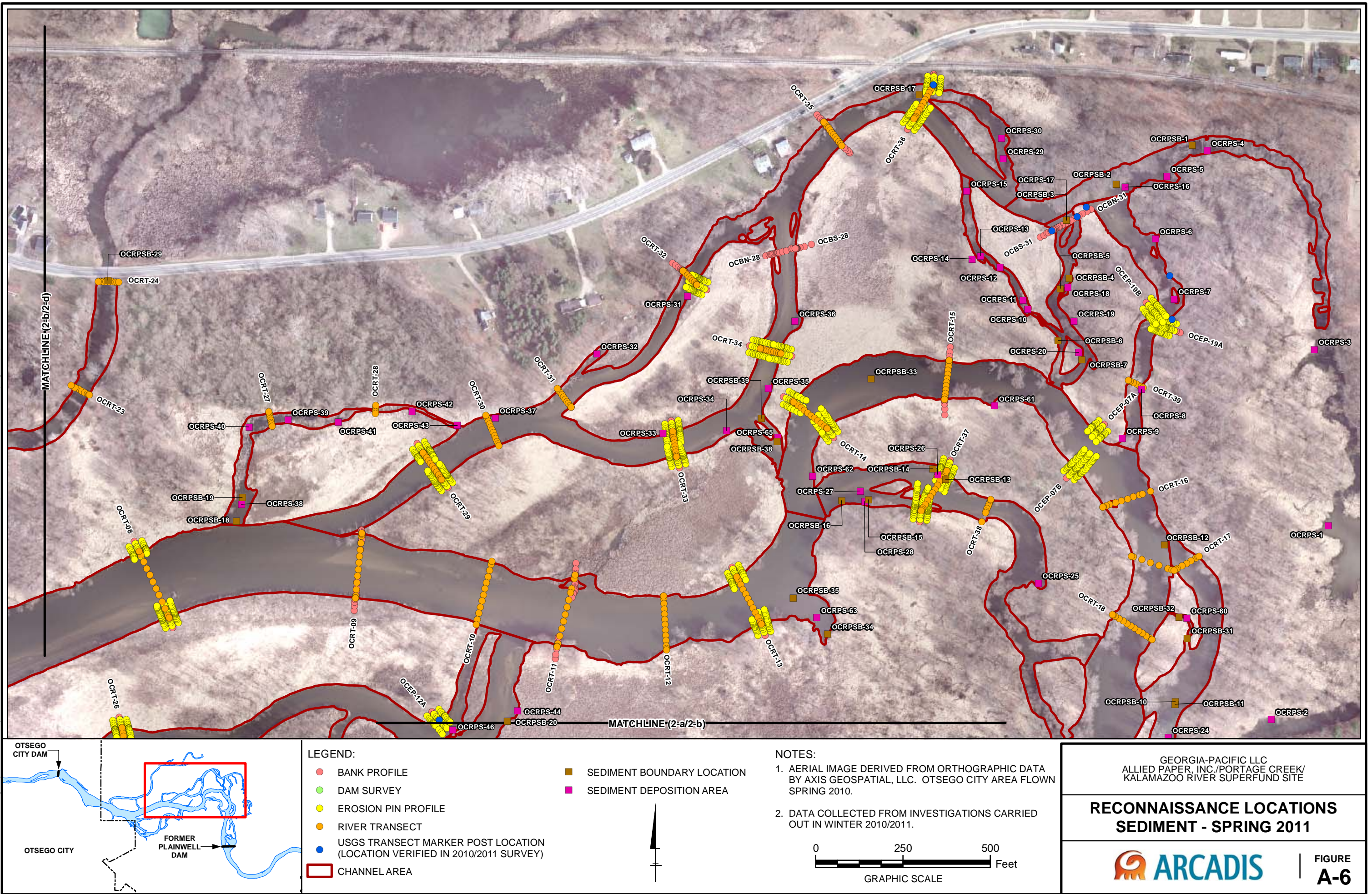
**FIGURE
A-2**

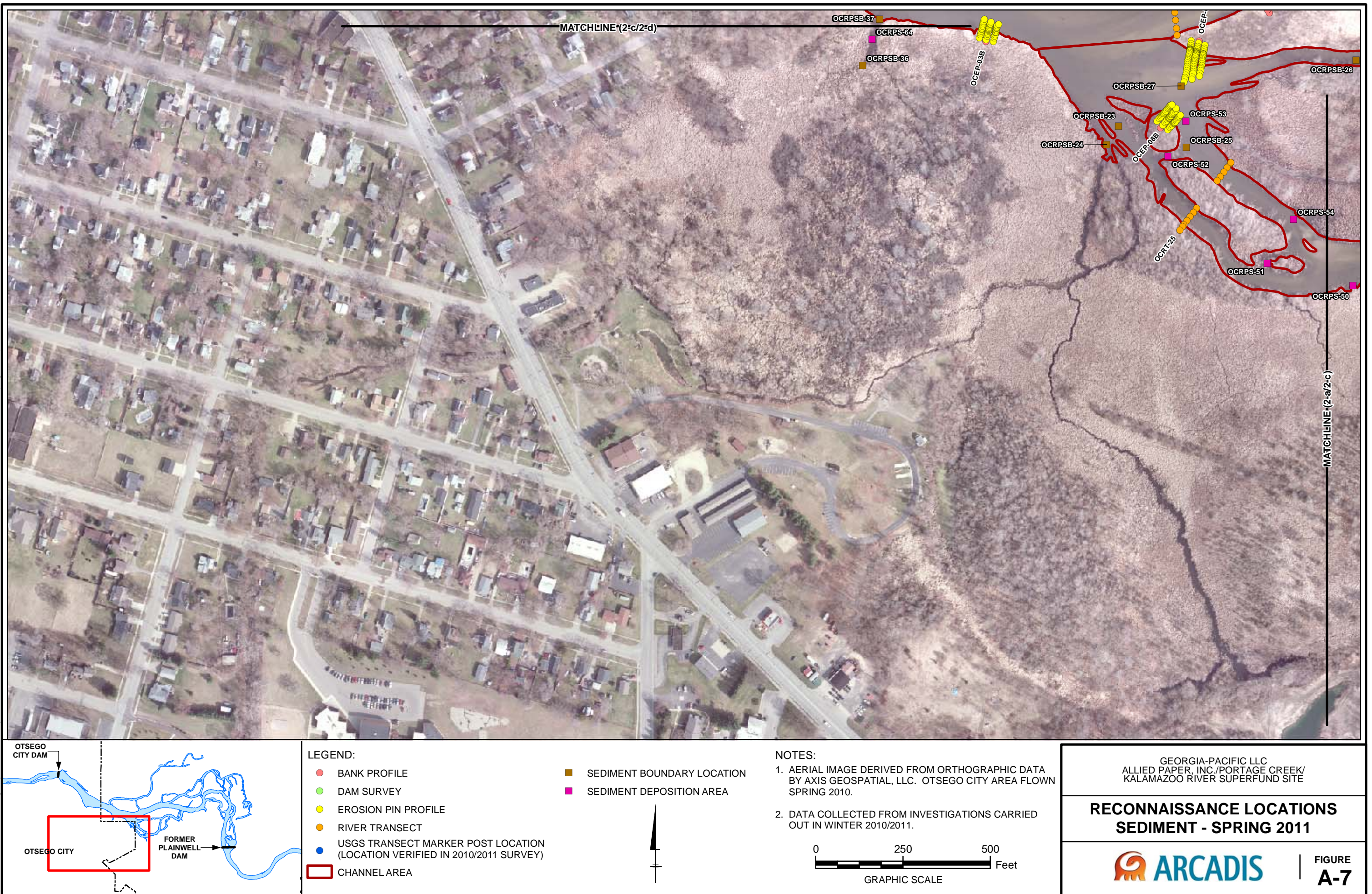






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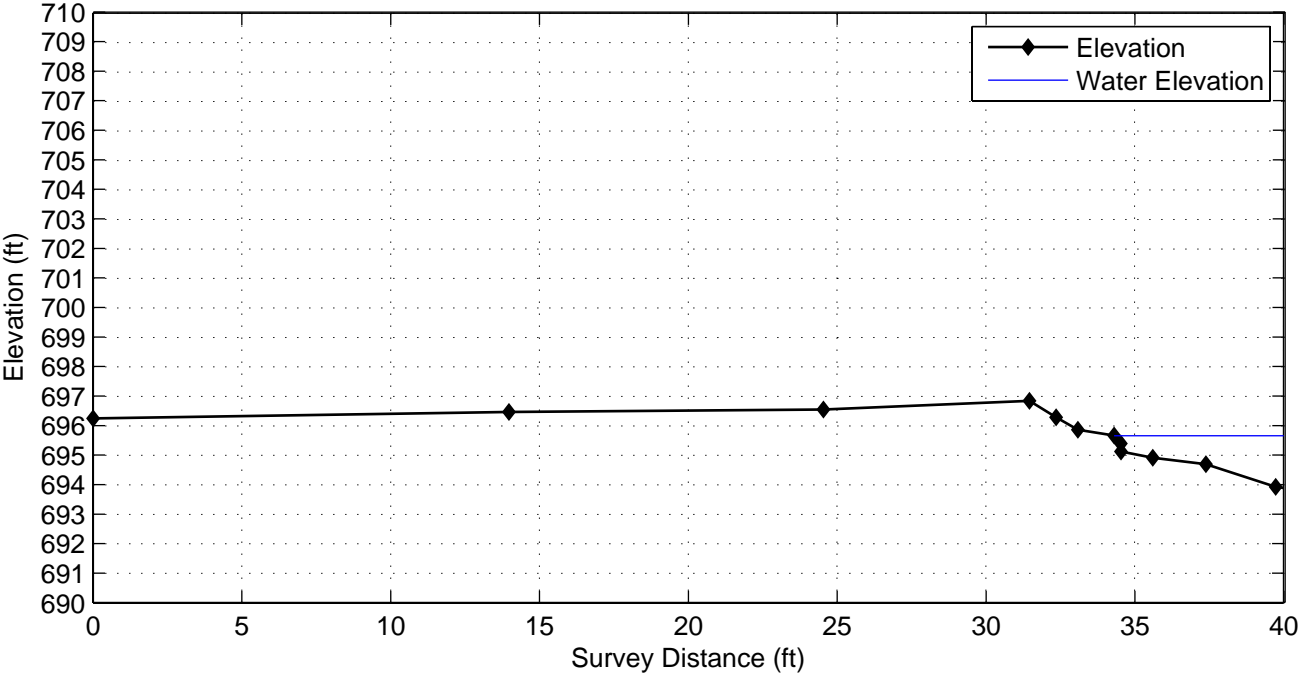
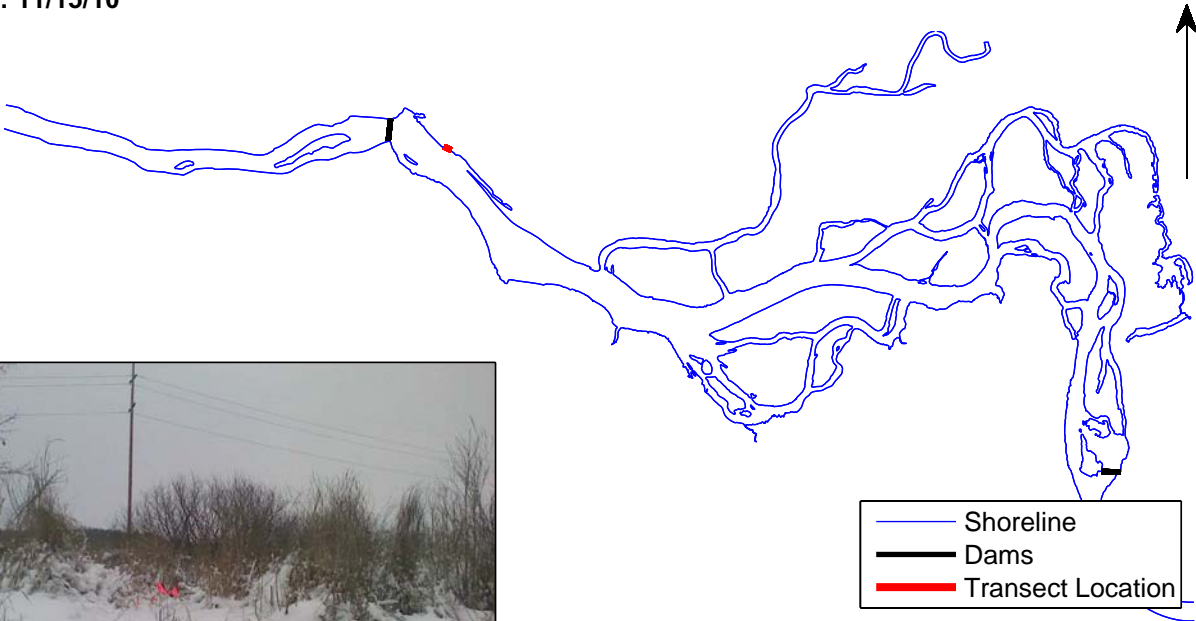


Attachment B

Bank Profile Survey and USGS
Transect Re-Survey Profiles

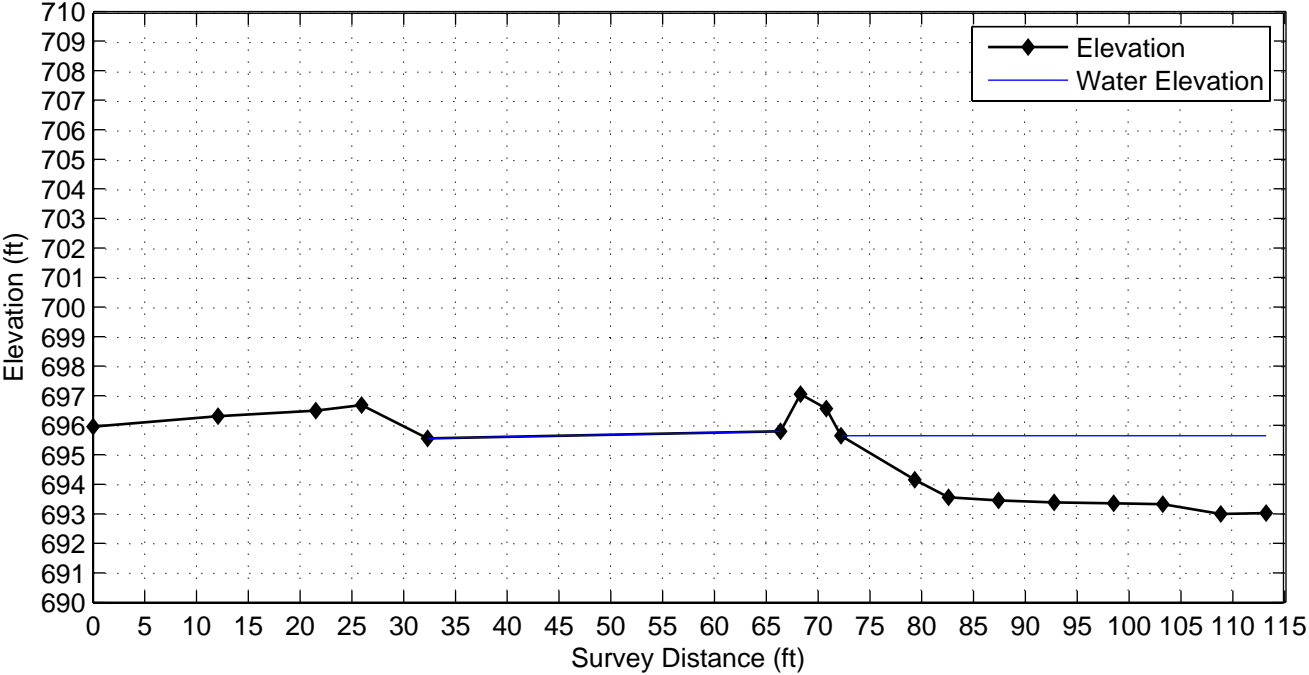
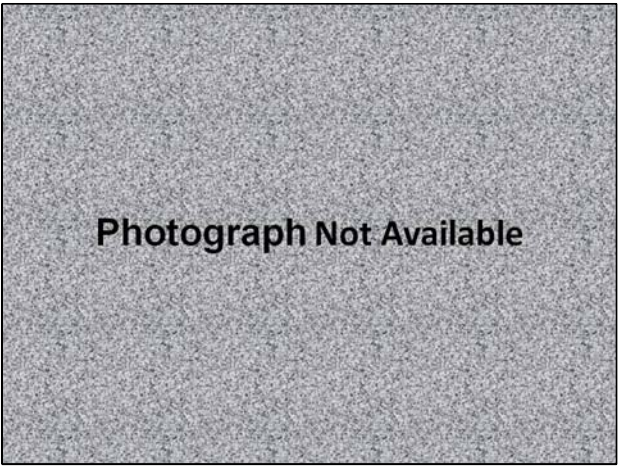
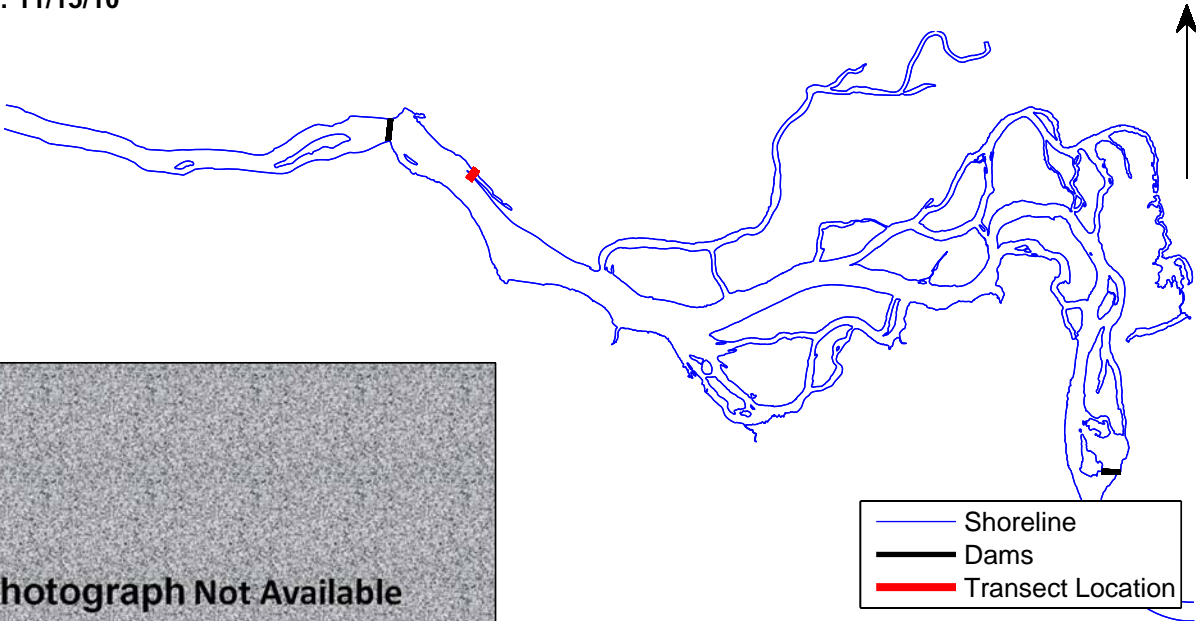
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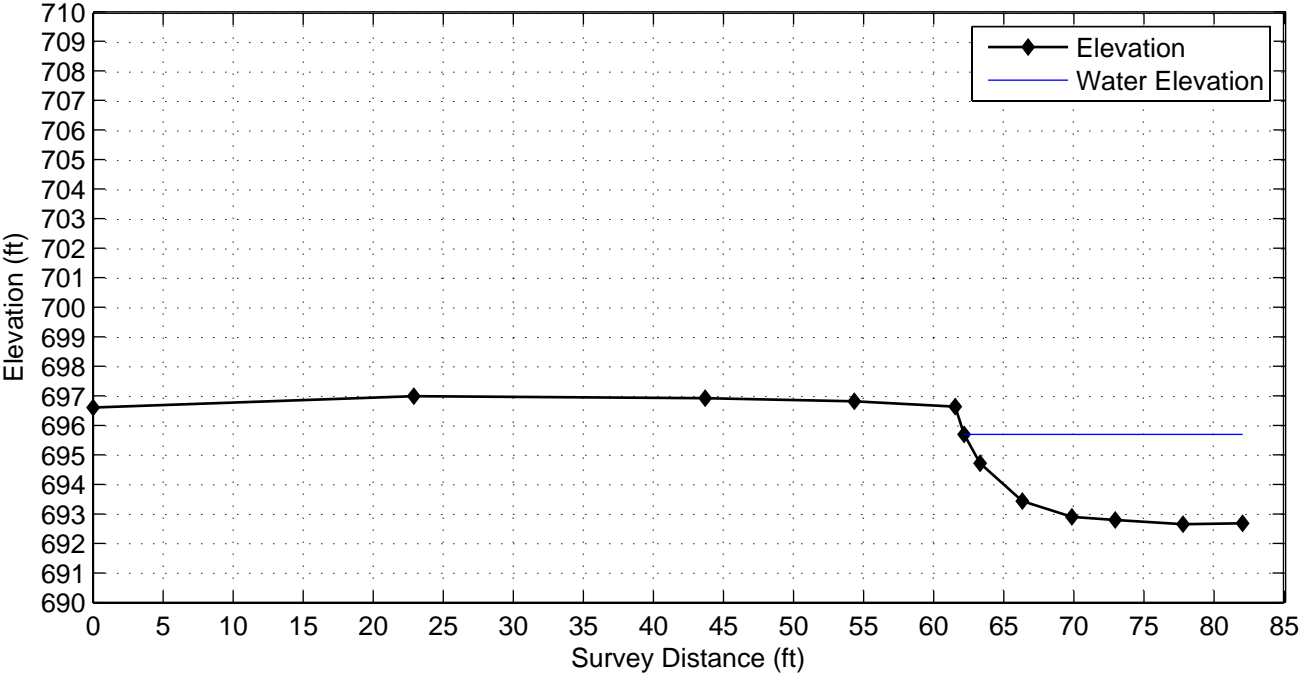
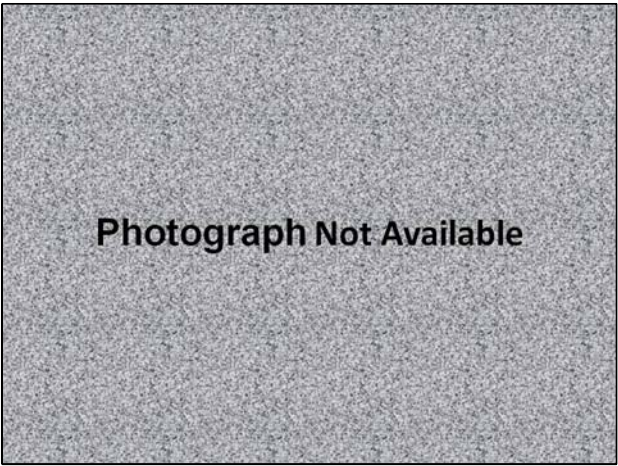
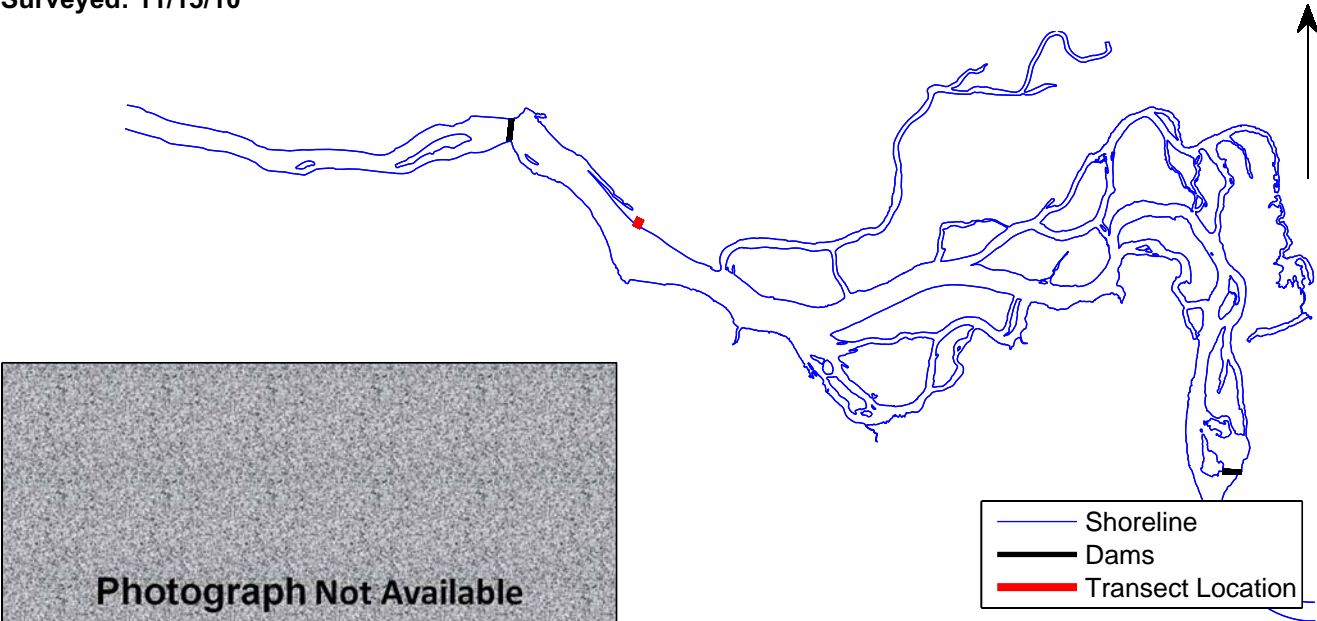
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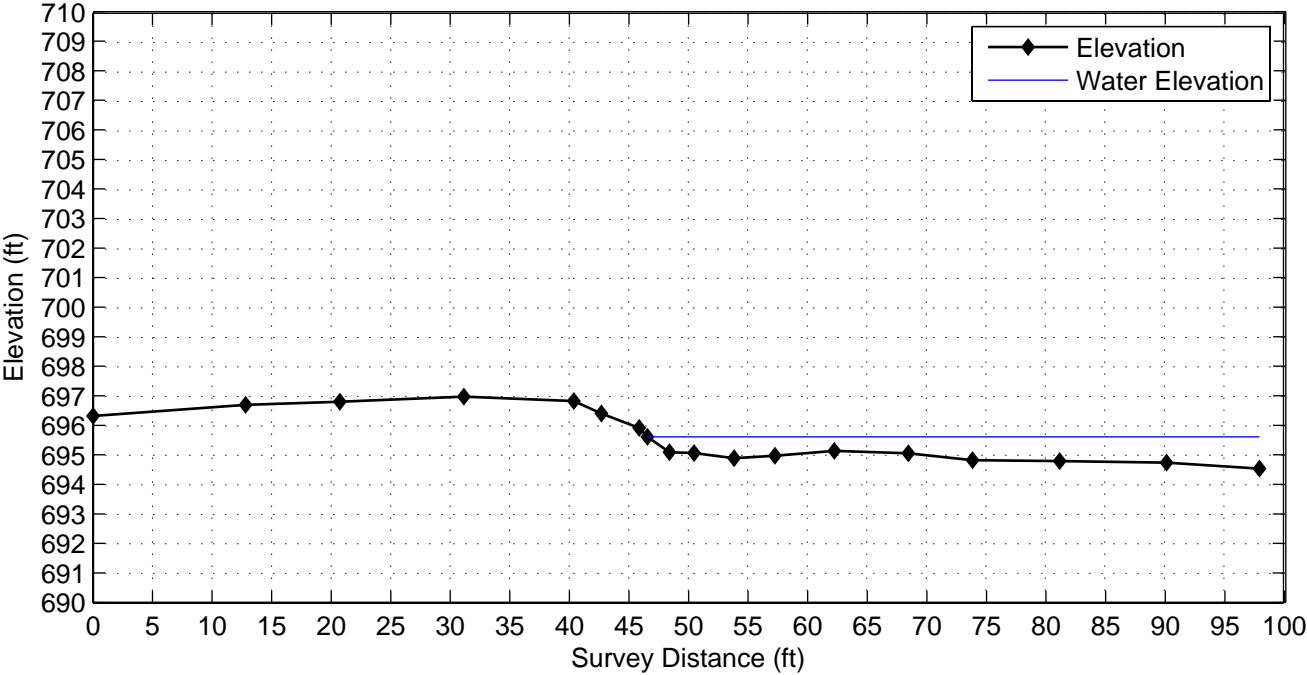
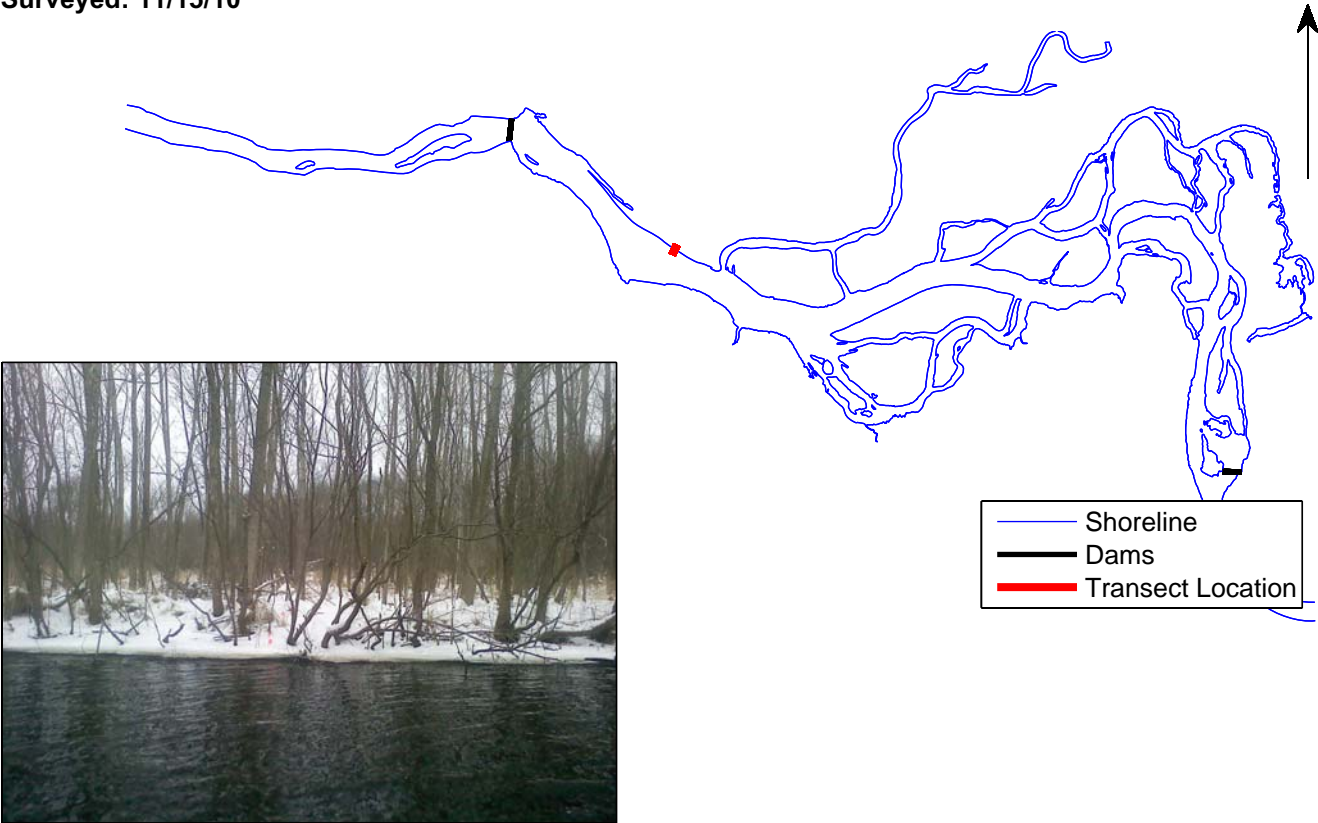
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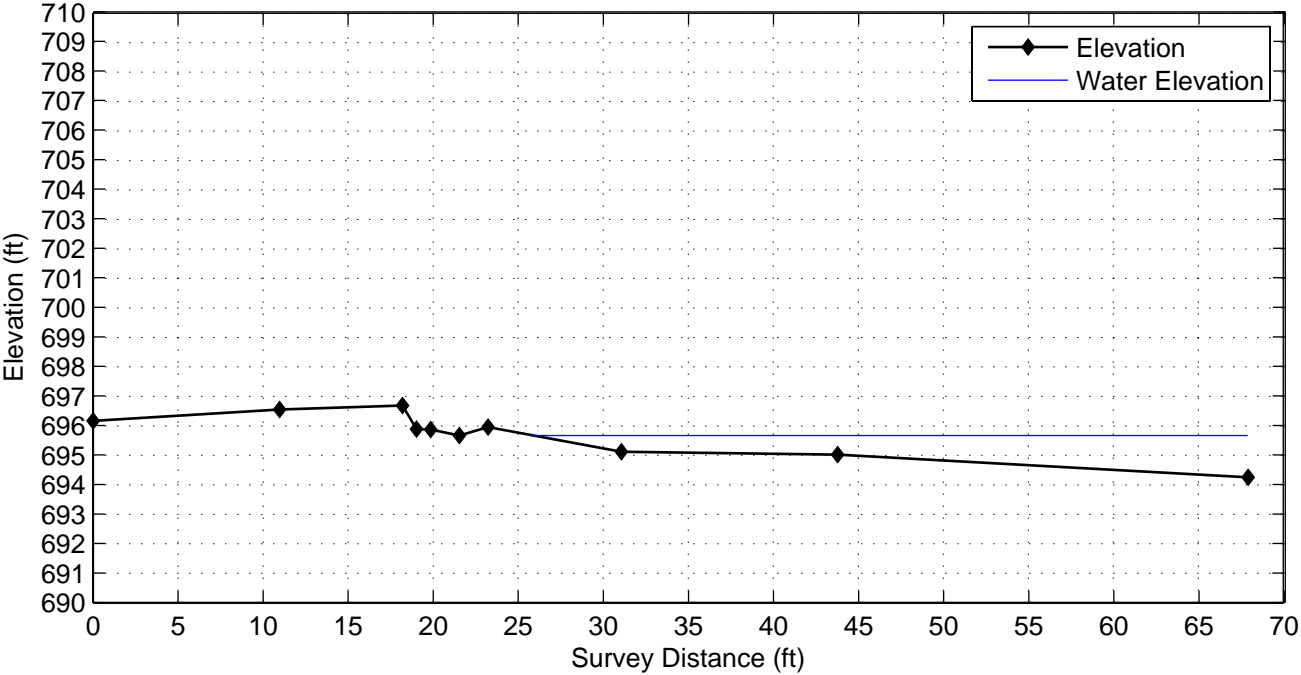
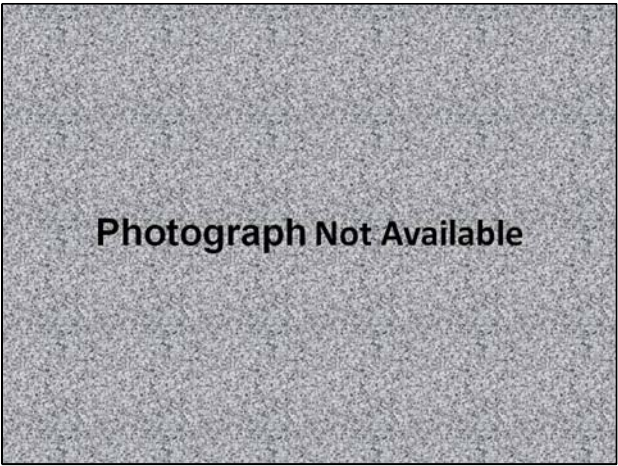
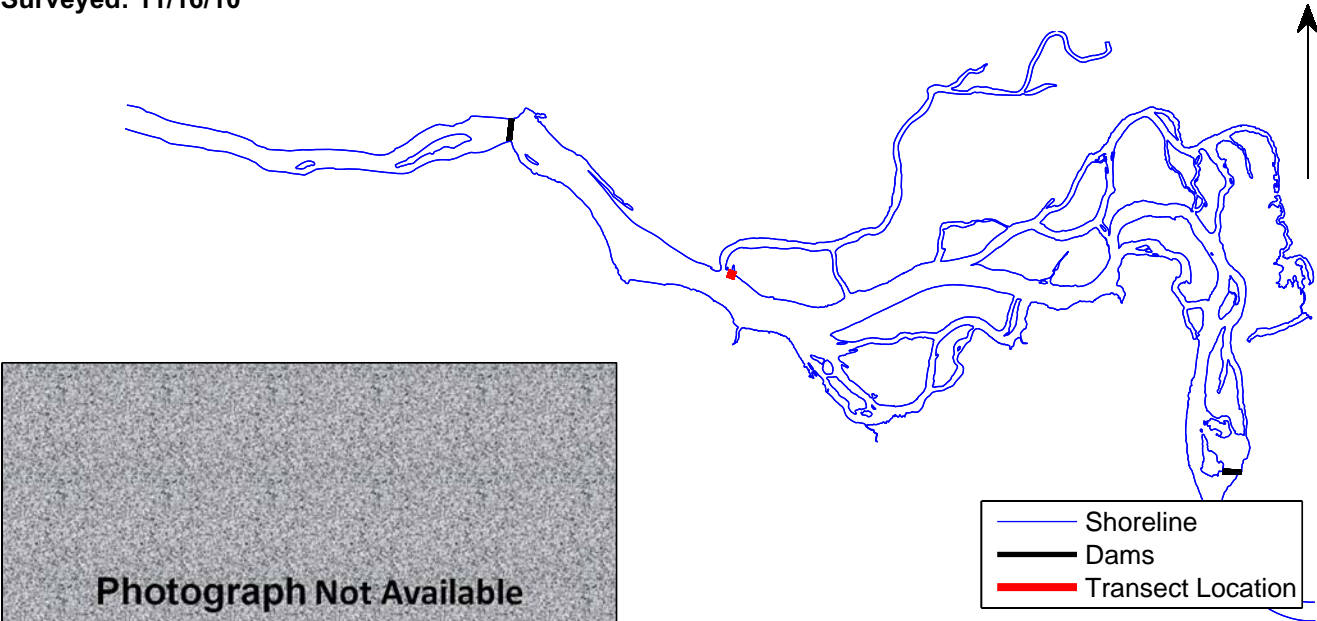
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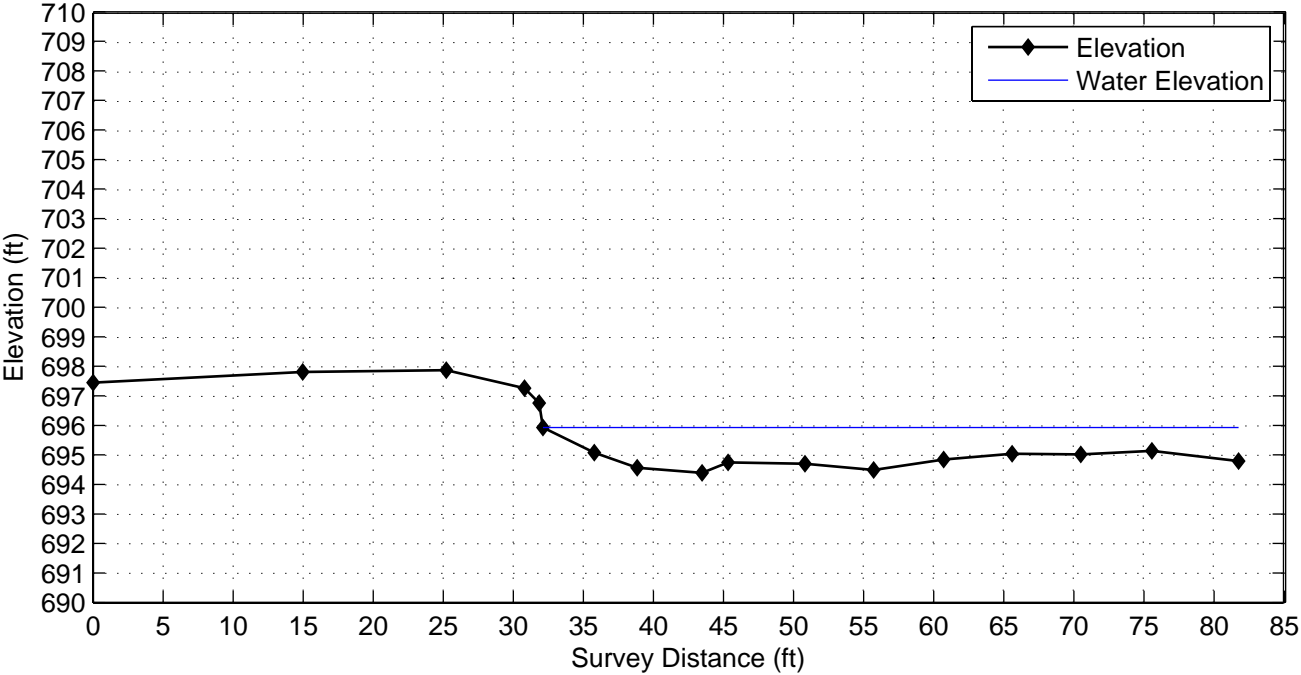
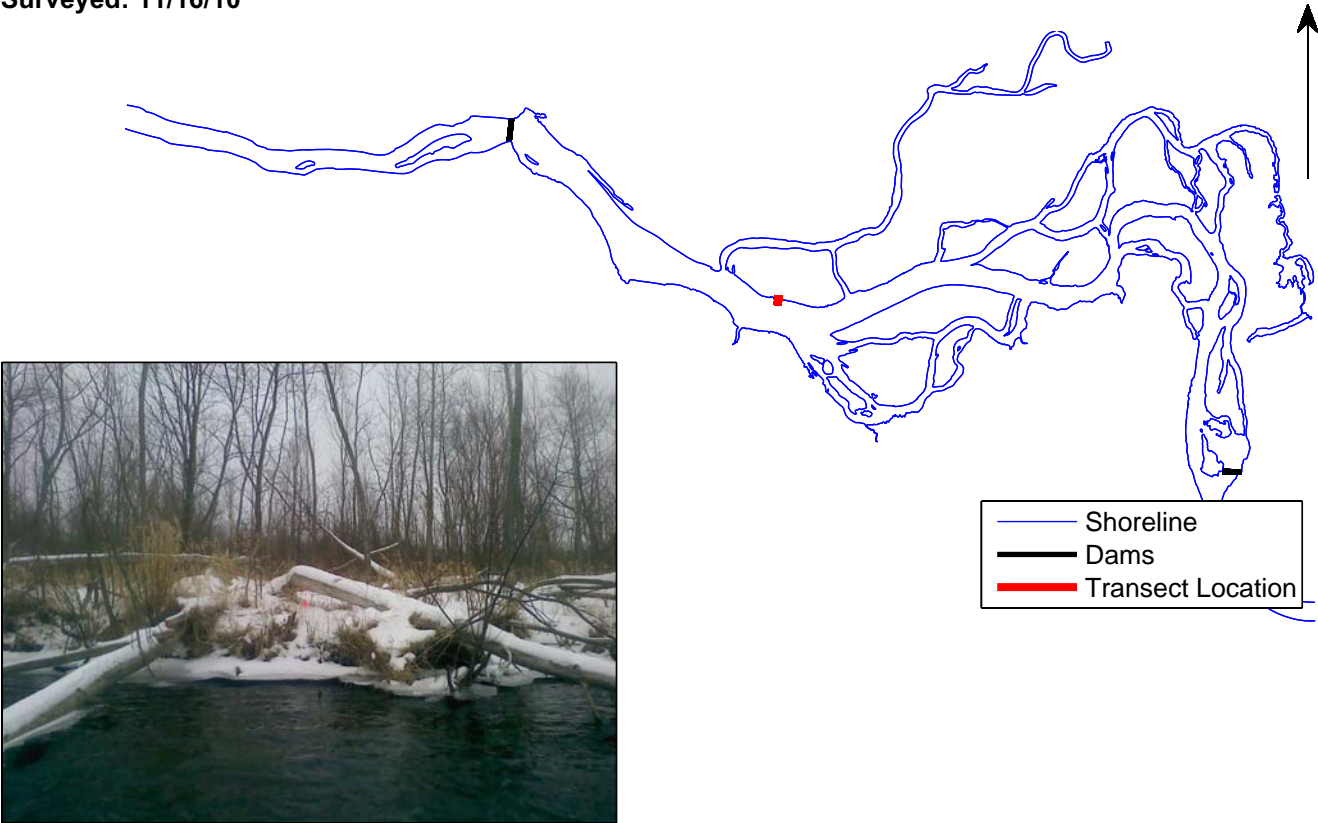
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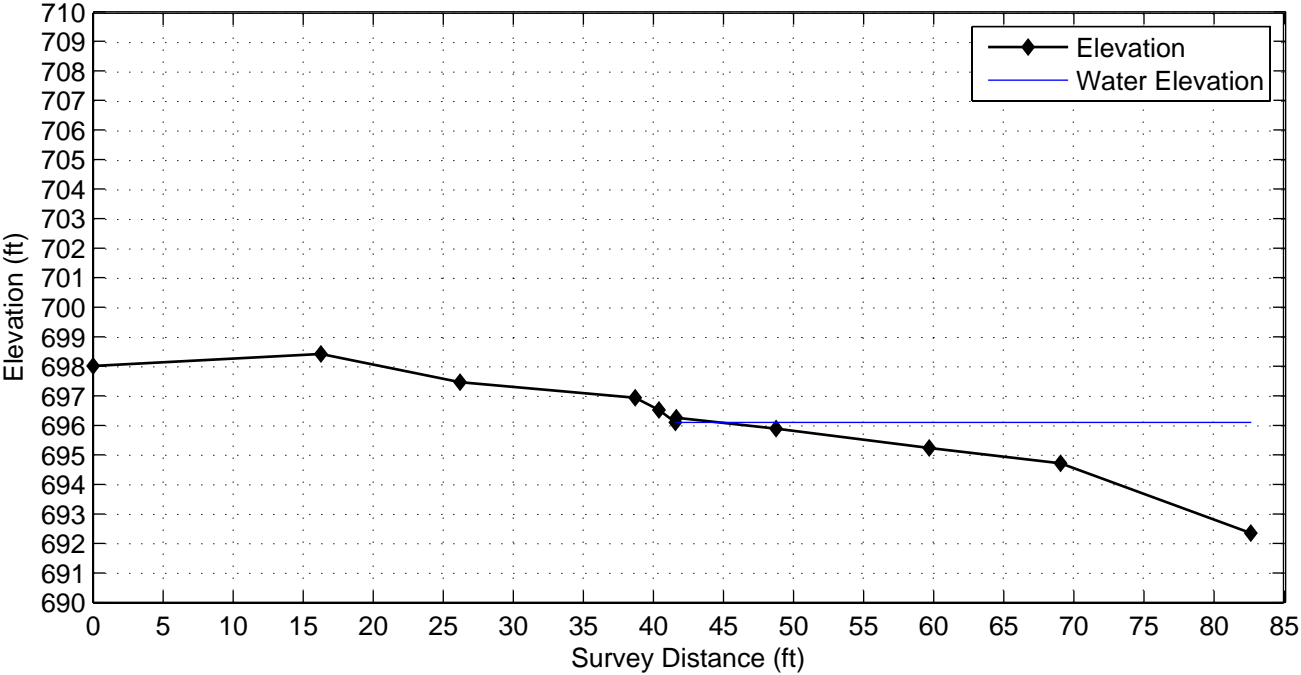
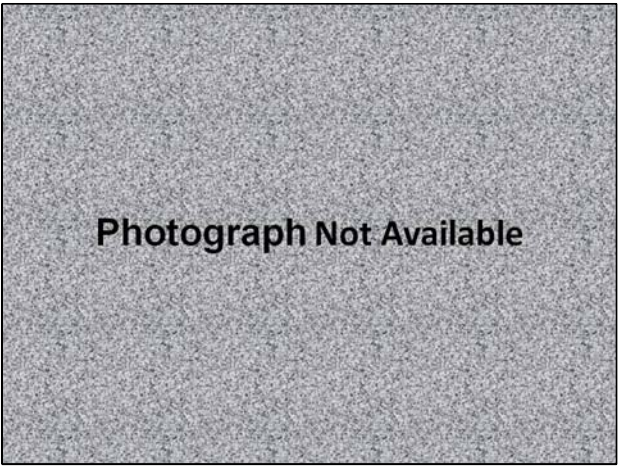
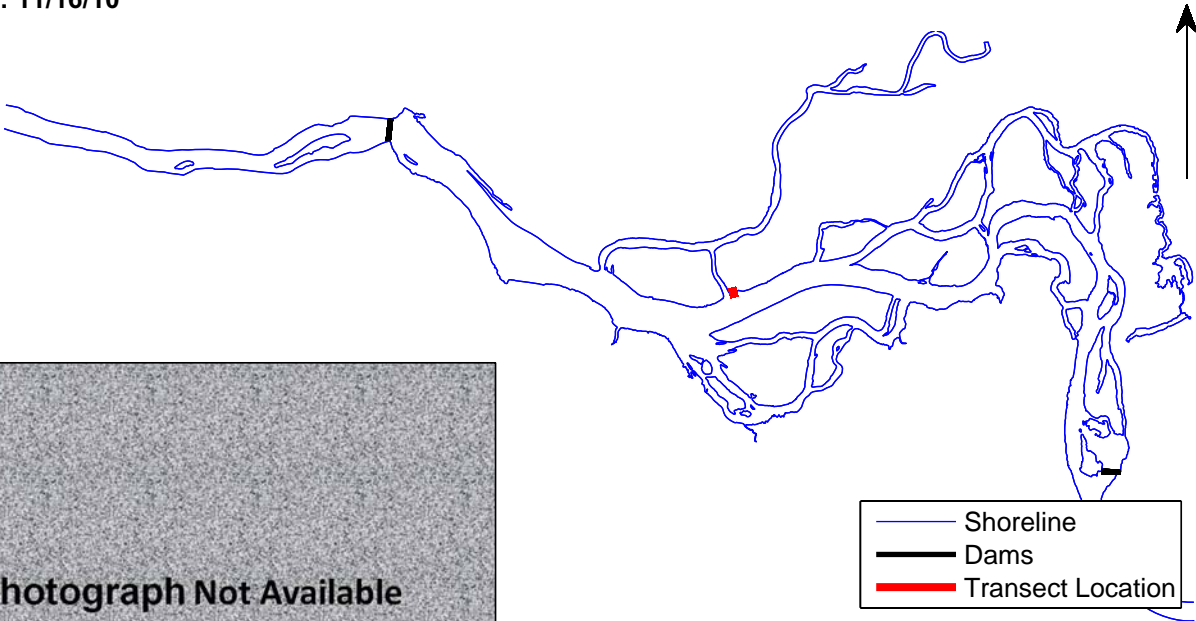
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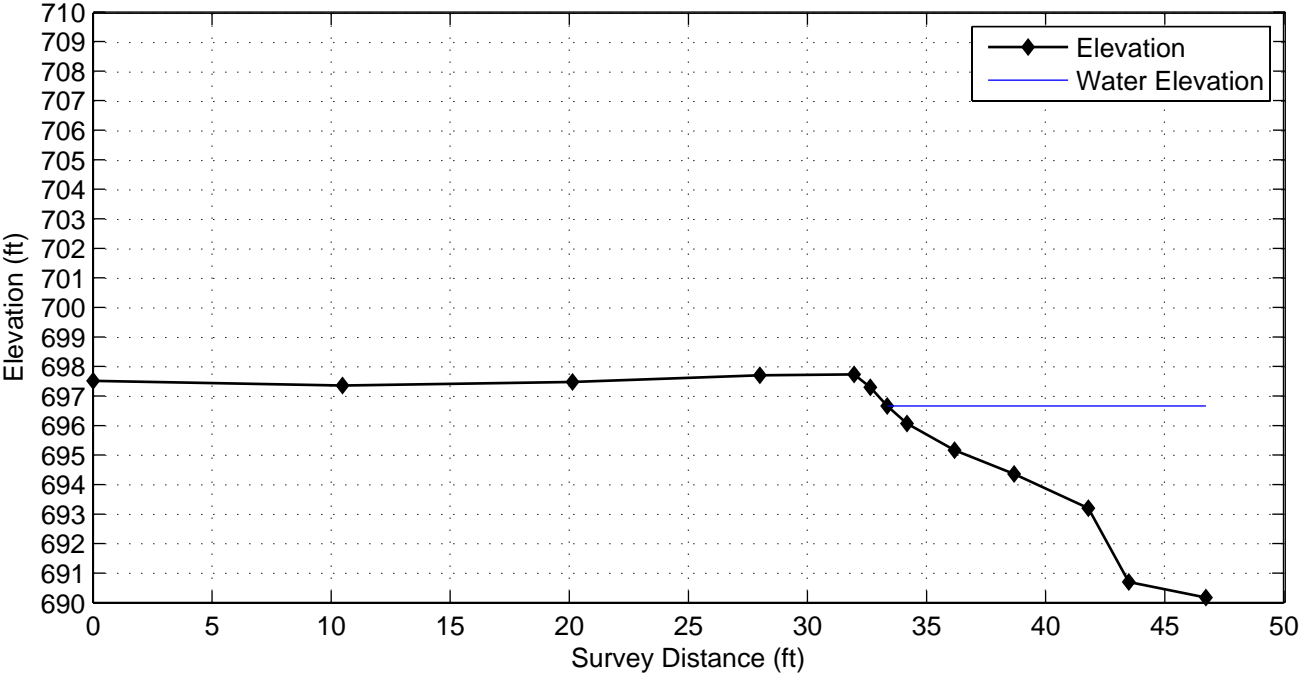
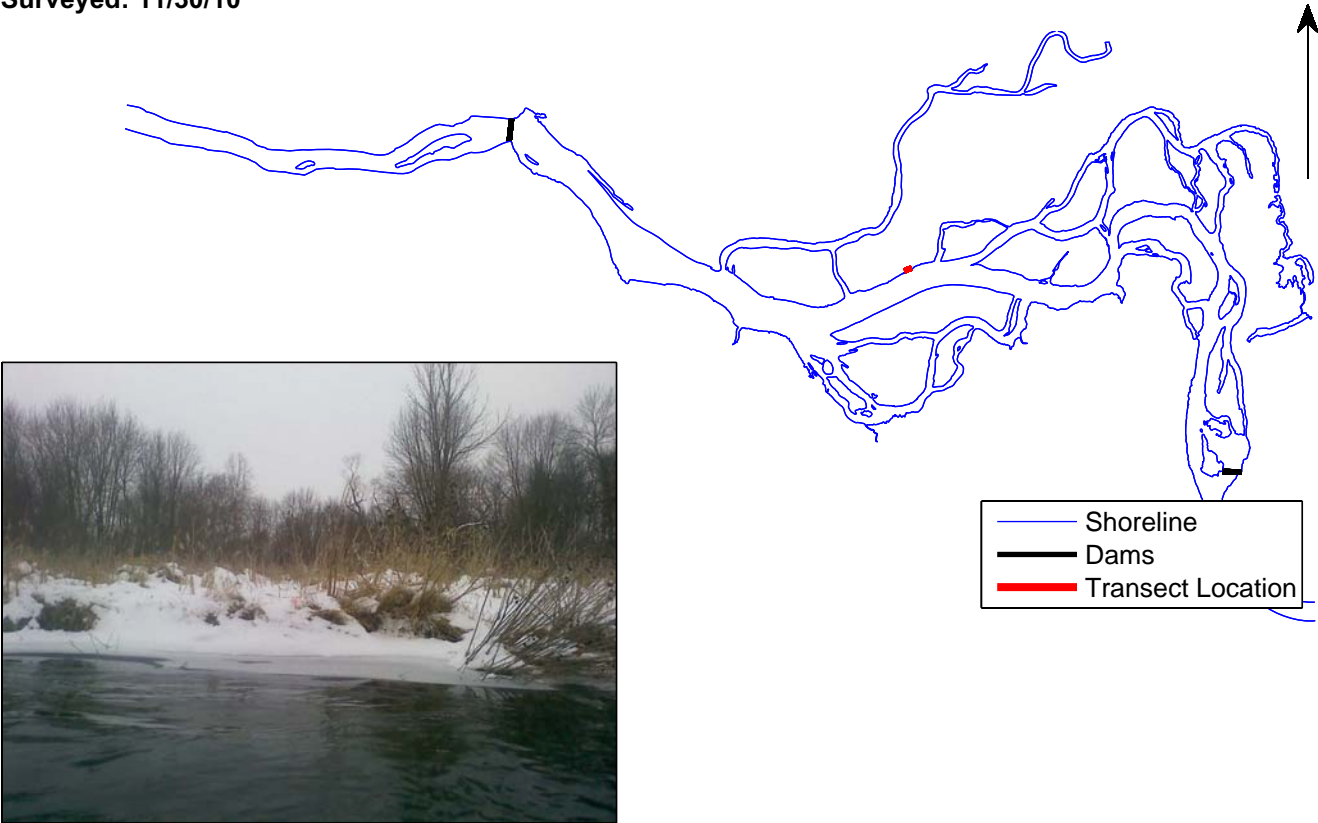
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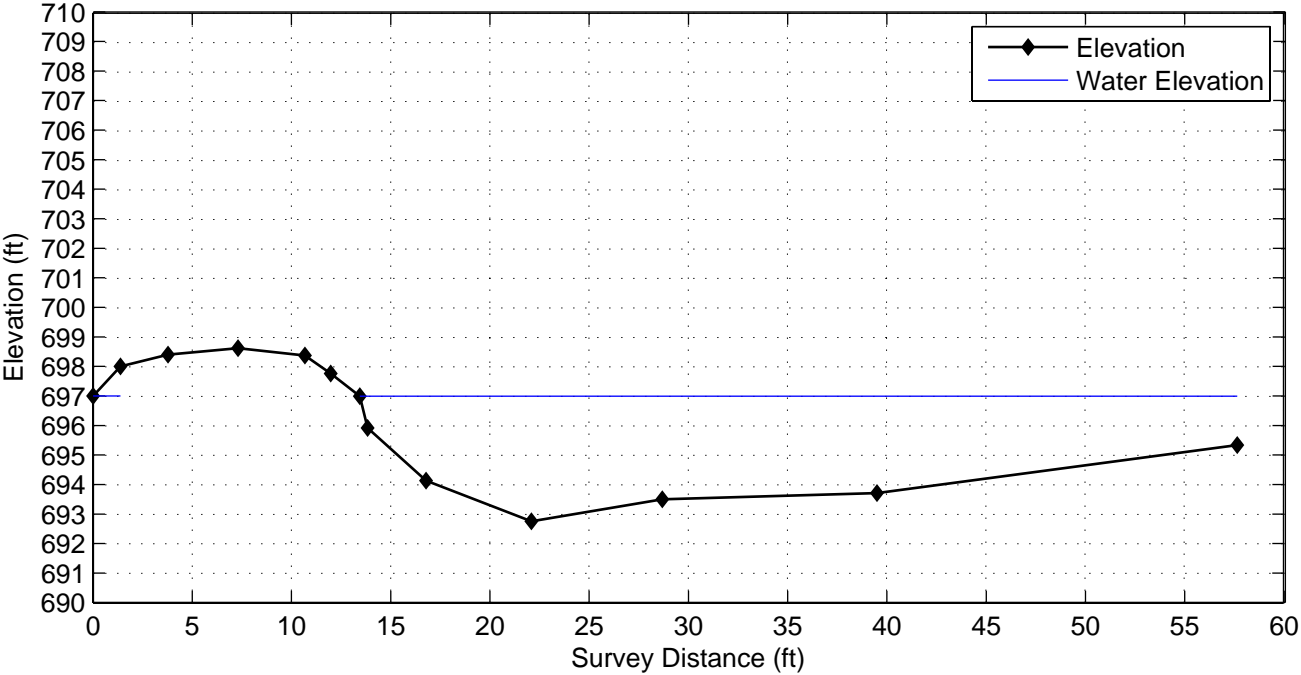
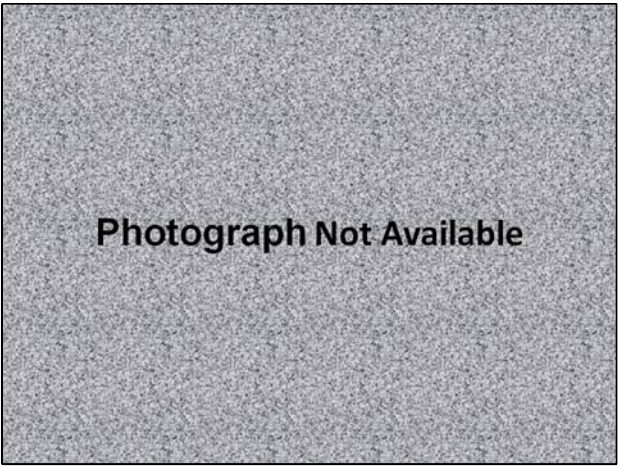
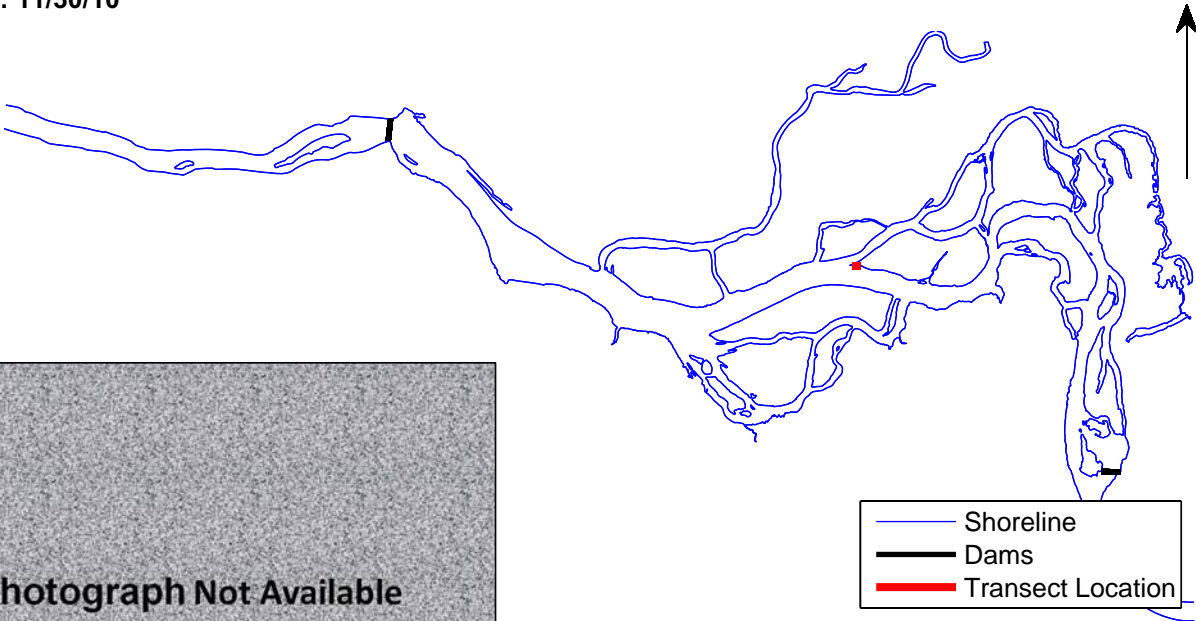
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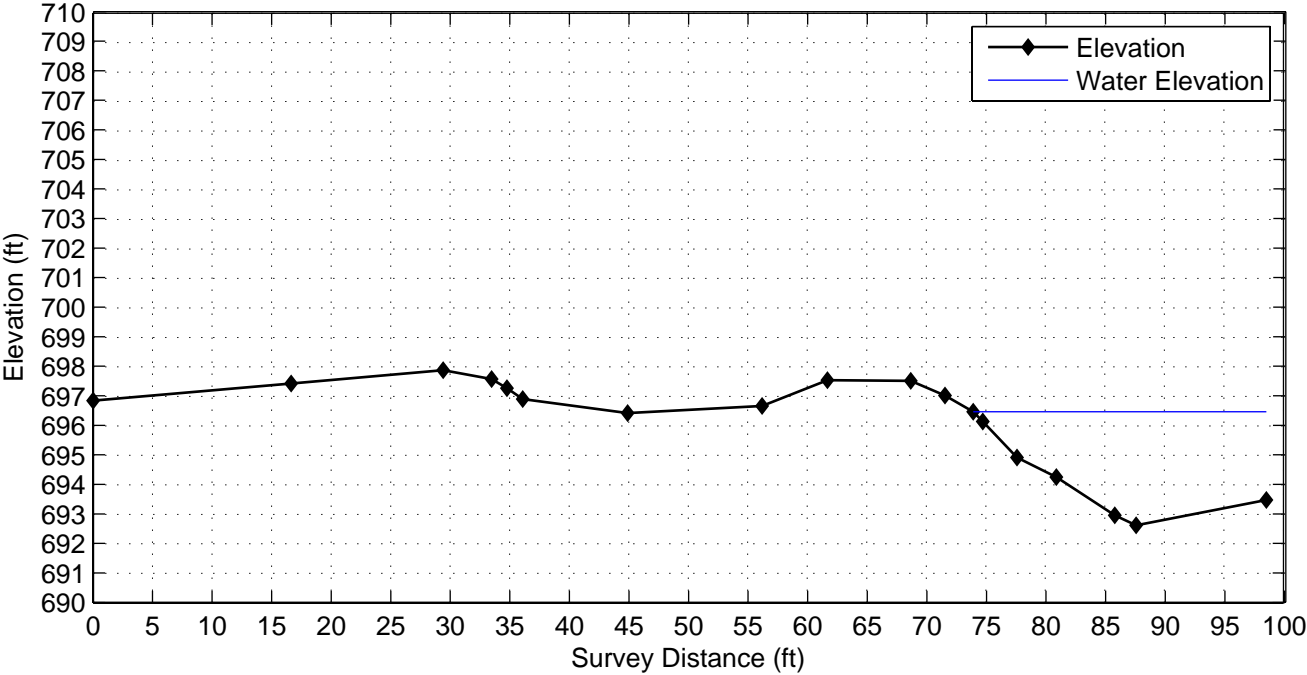
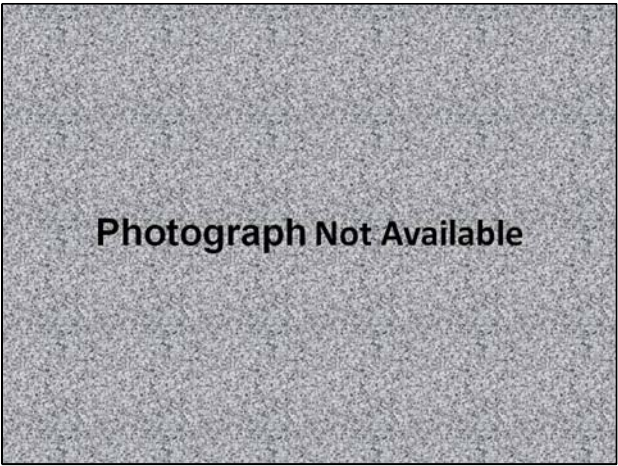
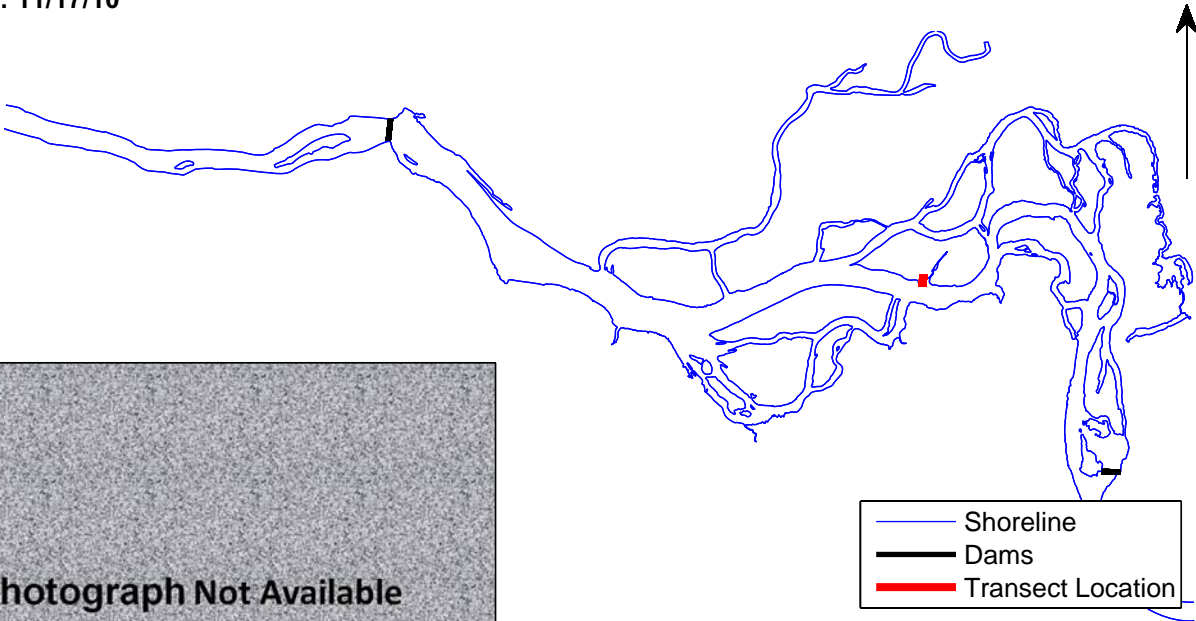
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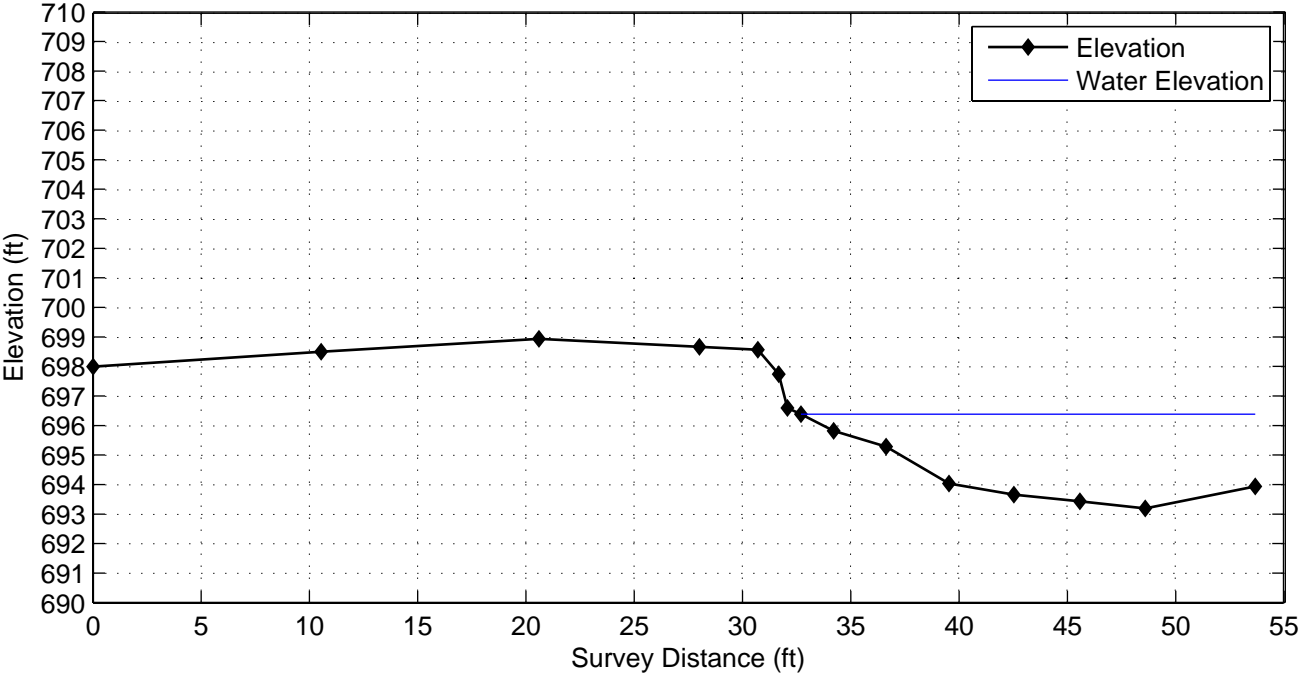
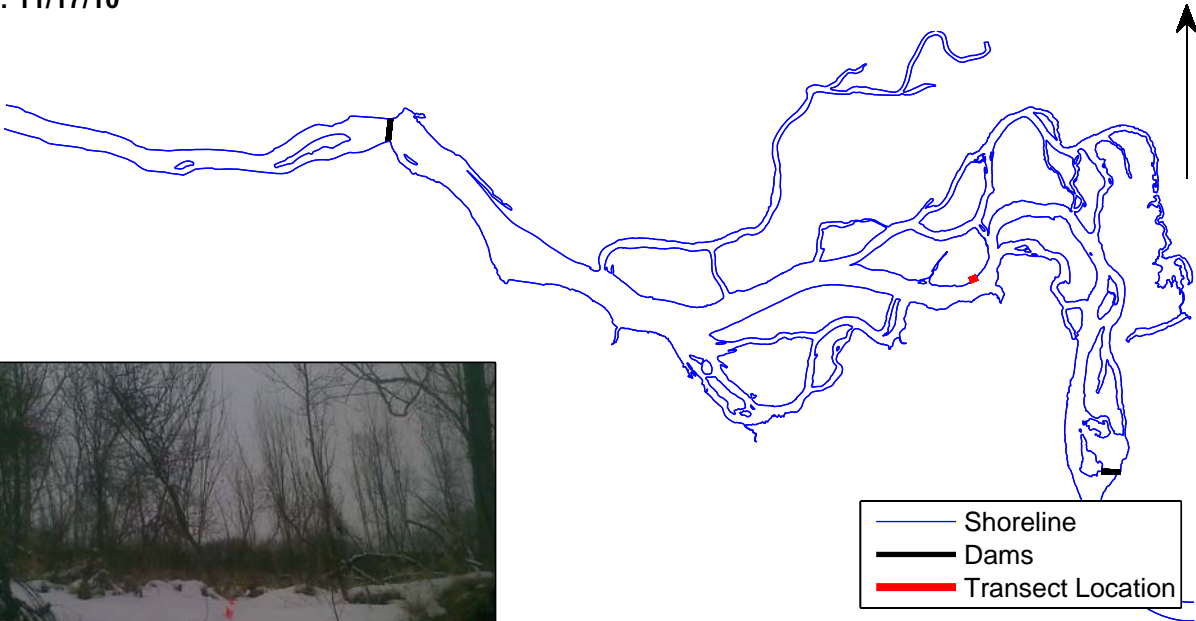
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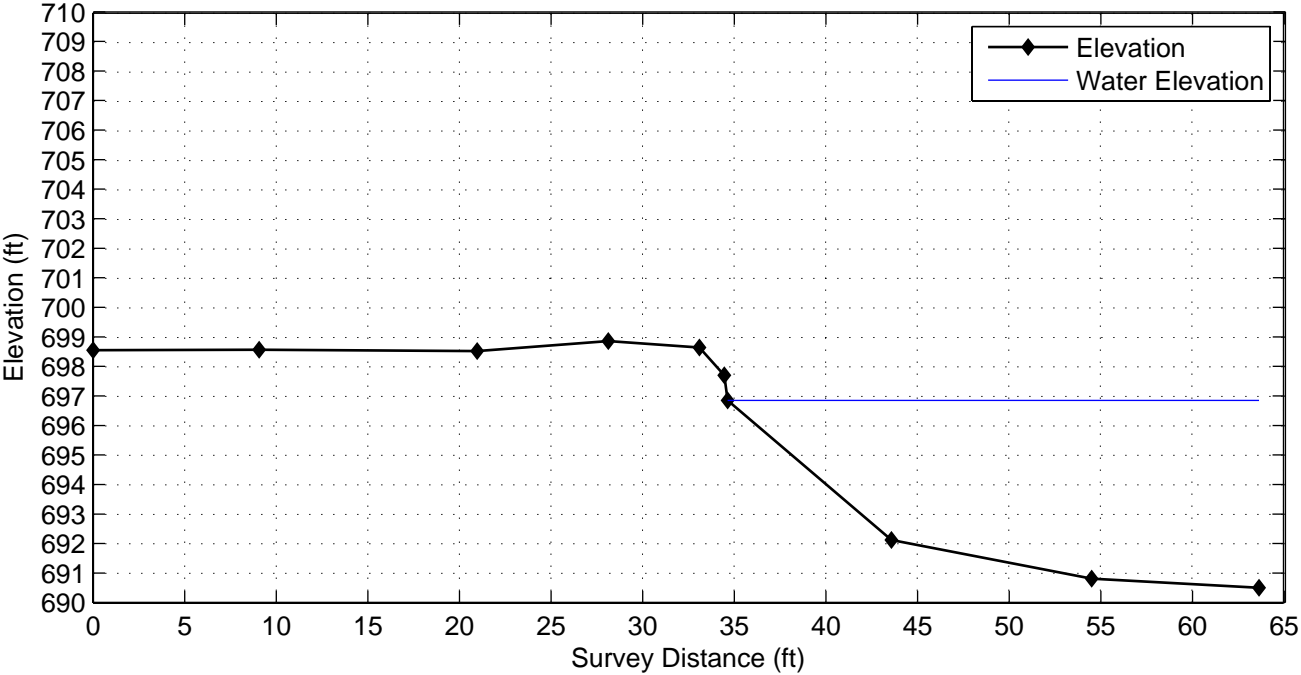
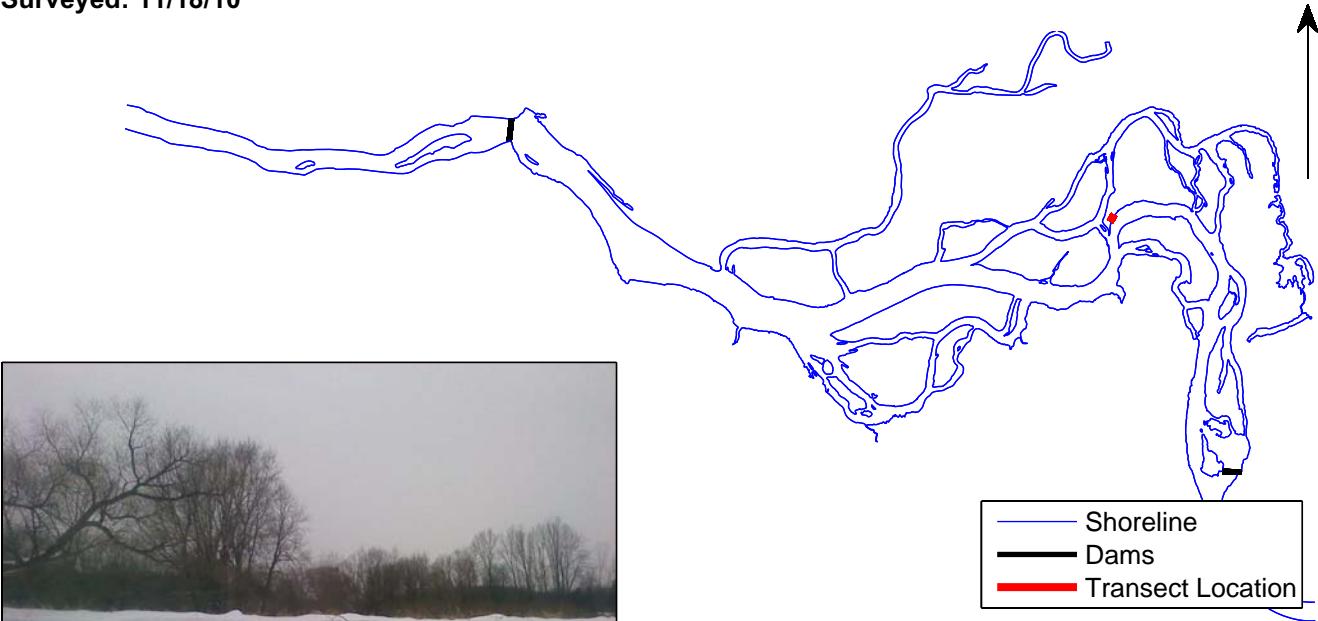
Otsego City Area 2 Bank Profiles
Transect: OCBN-11
Surveyed: 11/17/10

DRAFT



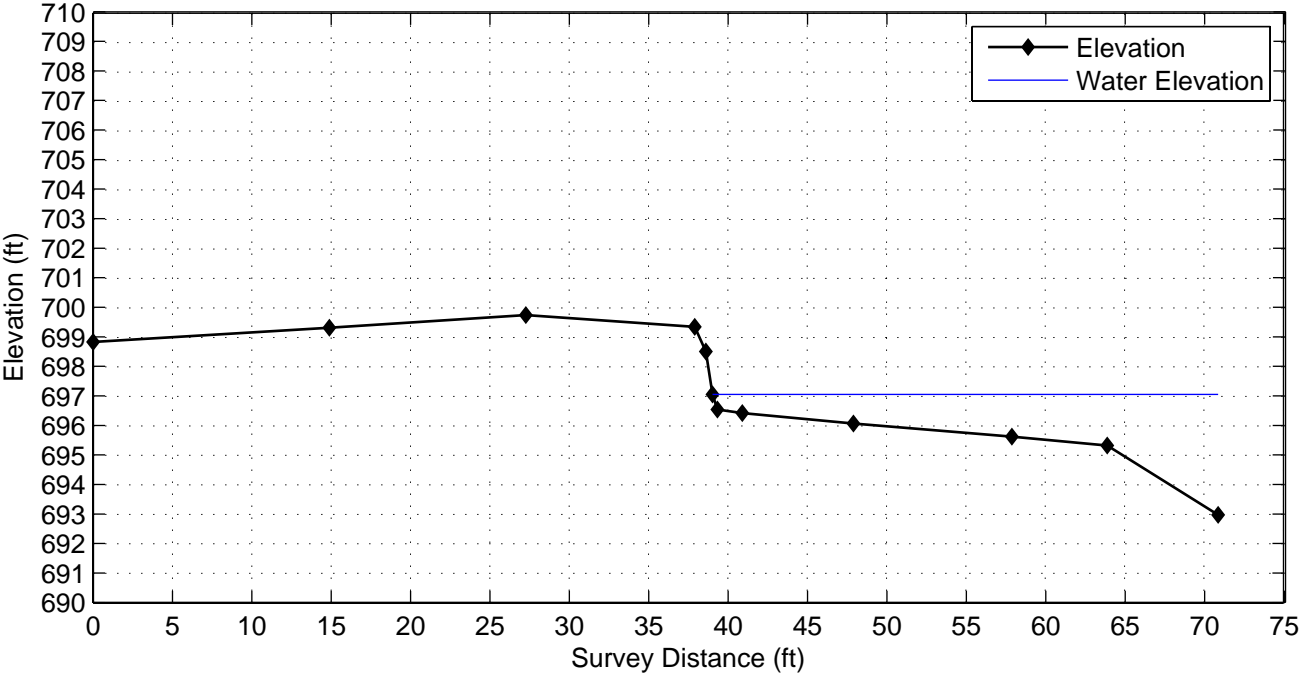
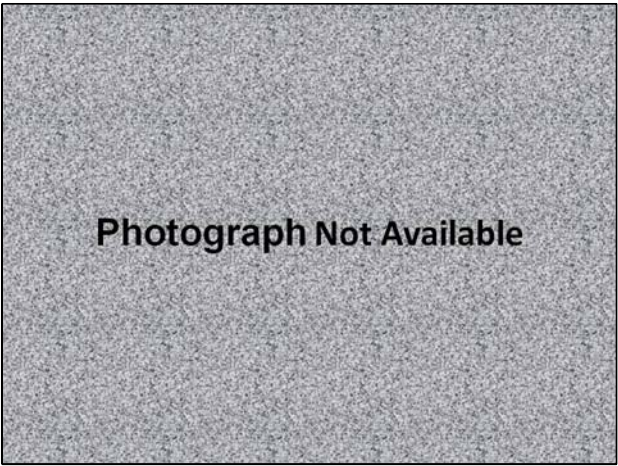
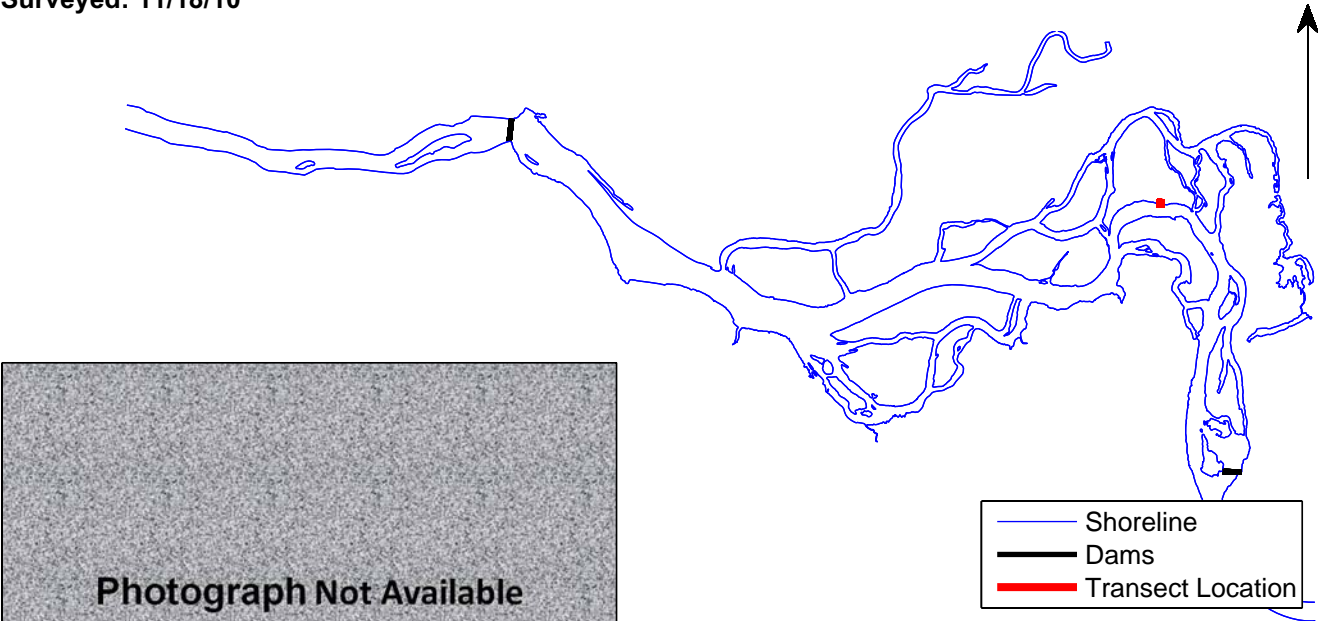
Otsego City Area 2 Bank Profiles
Transect: OCBN-12
Surveyed: 11/18/10

DRAFT



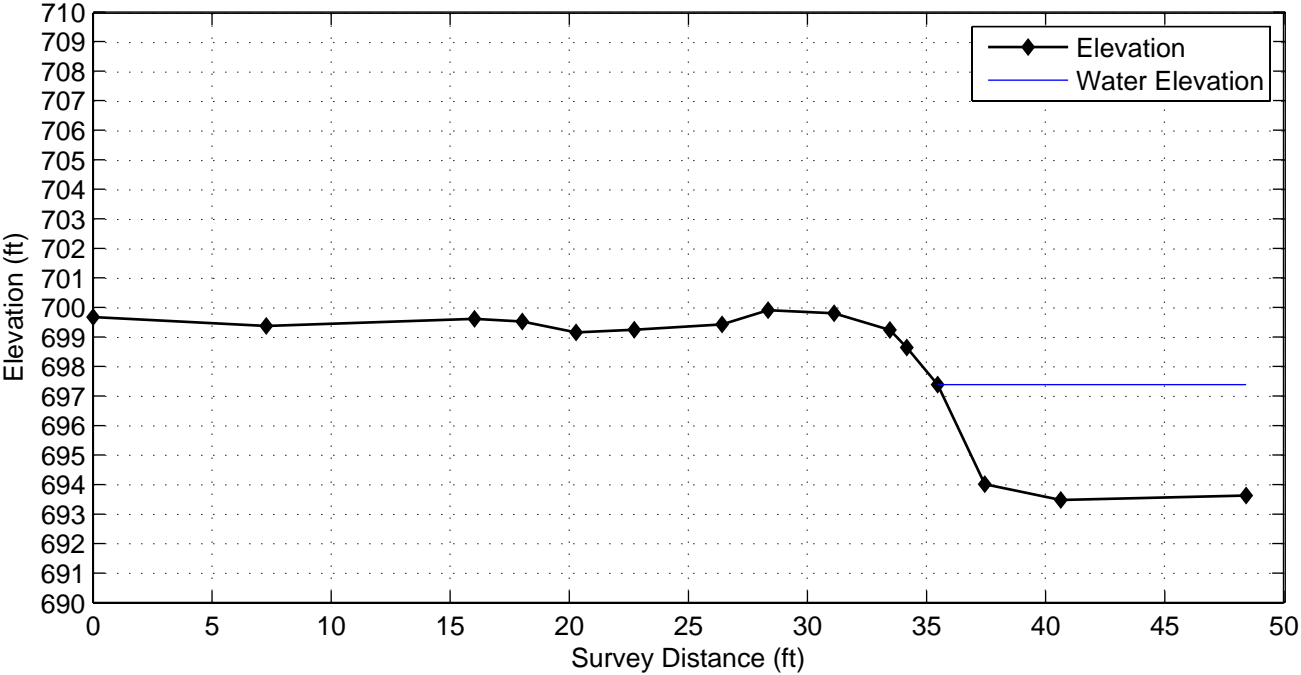
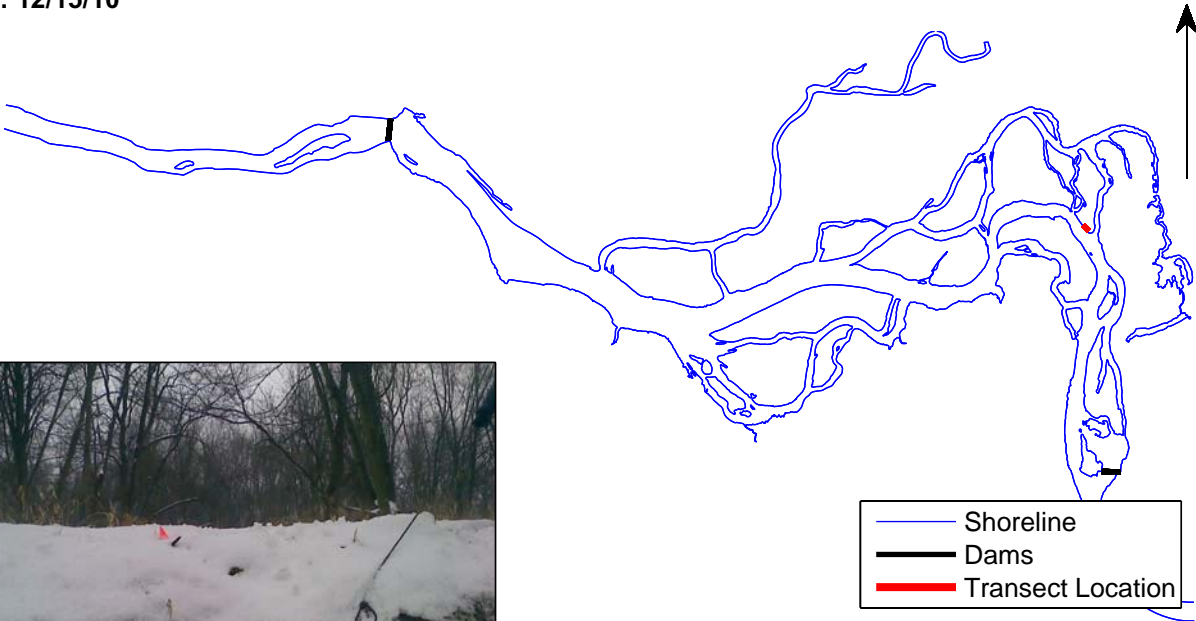
Otsego City Area 2 Bank Profiles
Transect: OCBN-13
Surveyed: 11/18/10

DRAFT



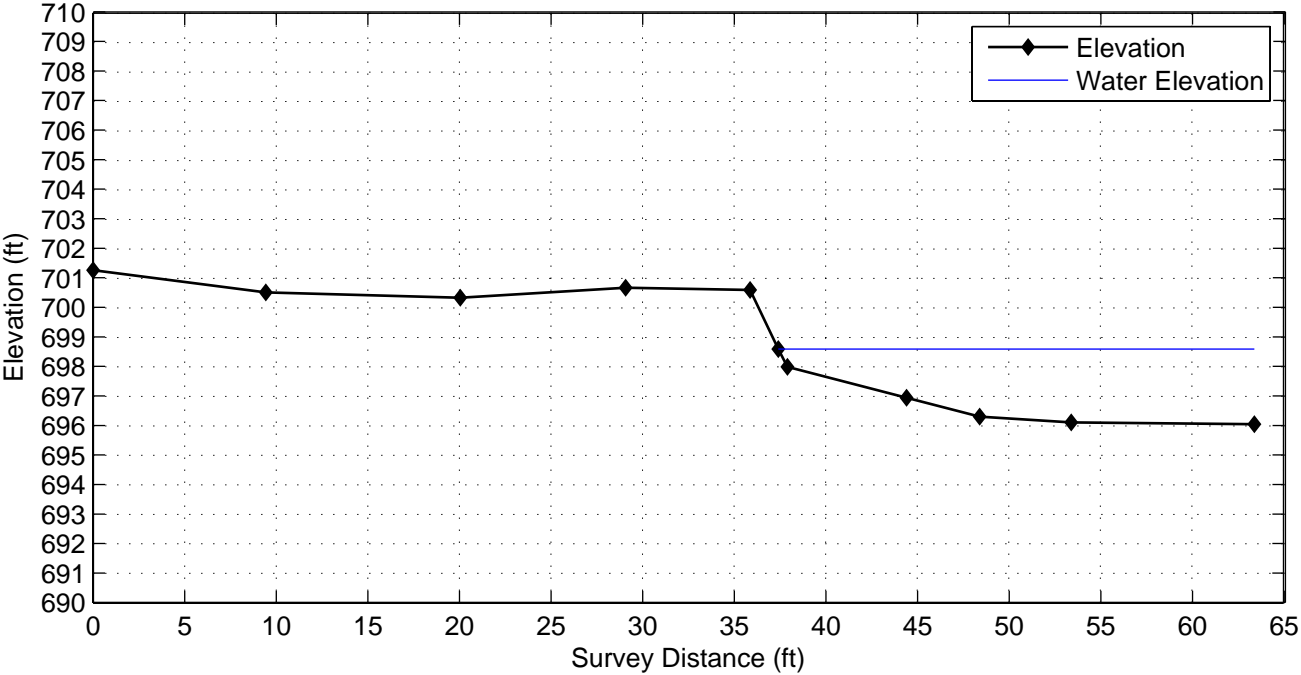
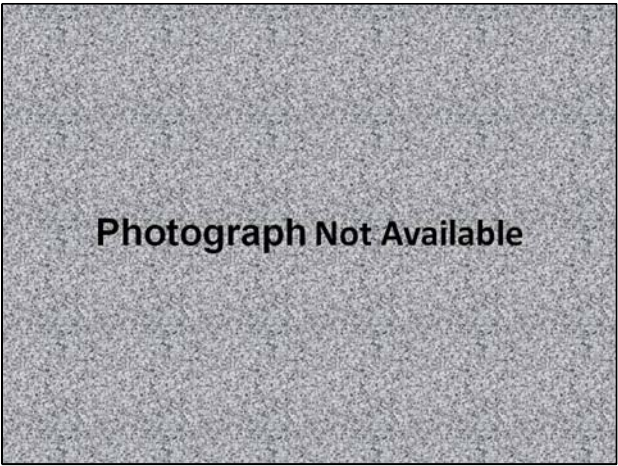
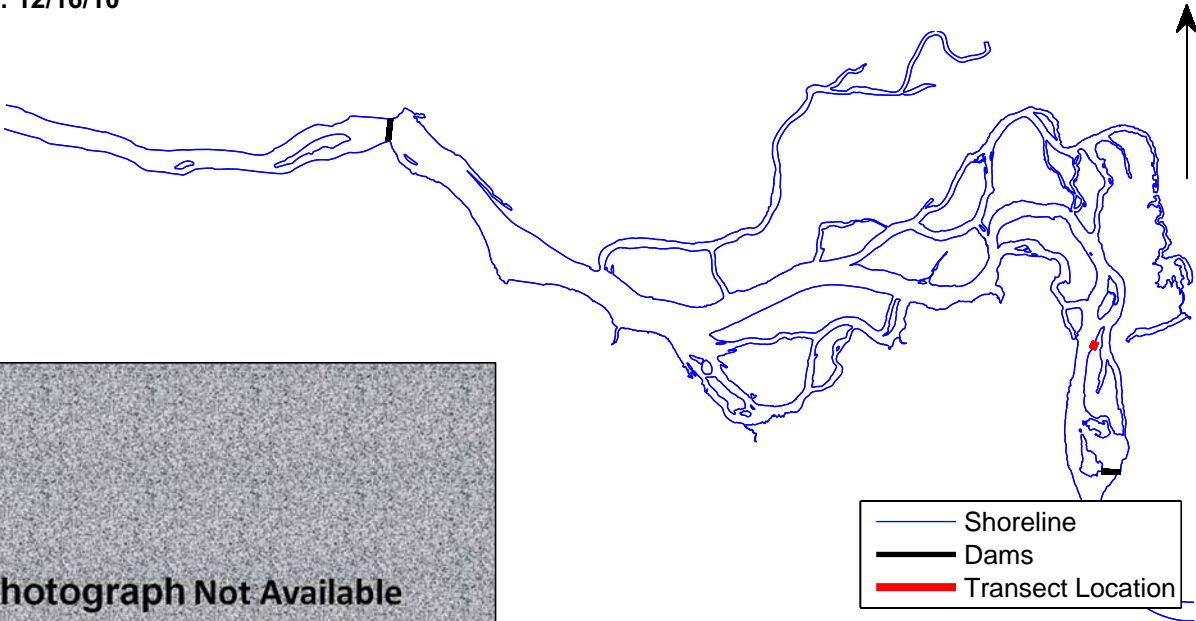
Otsego City Area 2 Bank Profiles
Transect: OCBN-14
Surveyed: 12/15/10

DRAFT



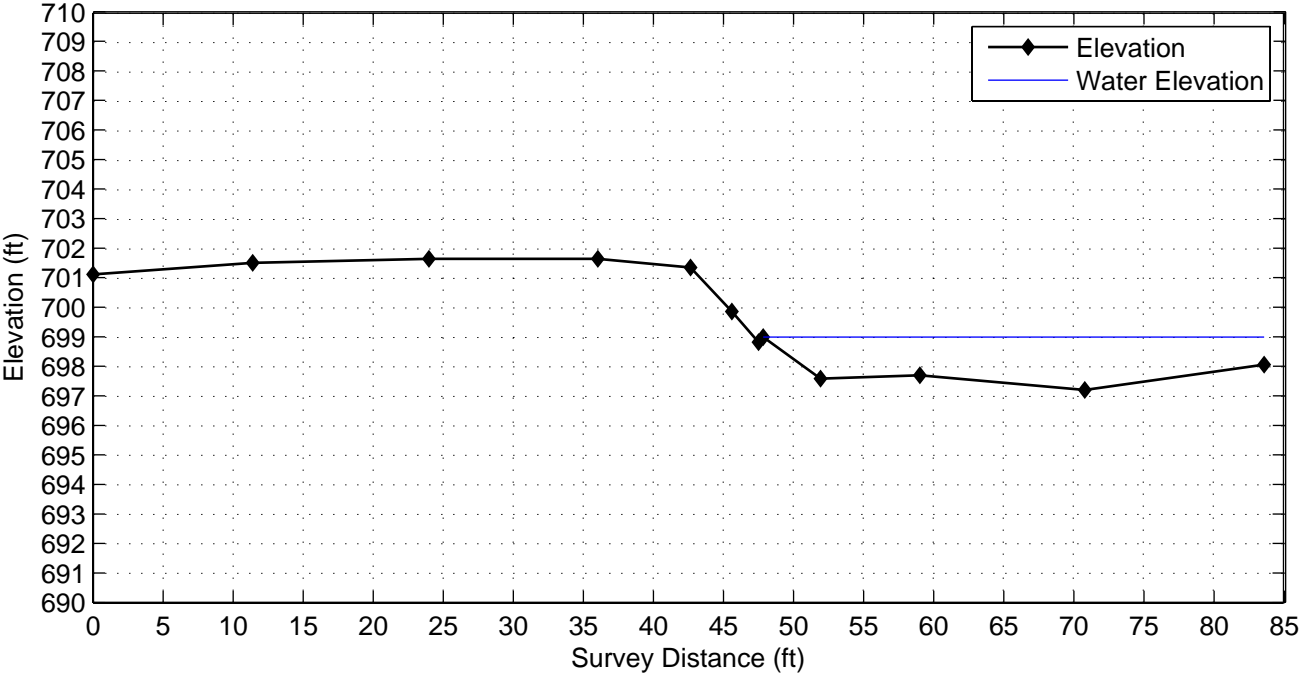
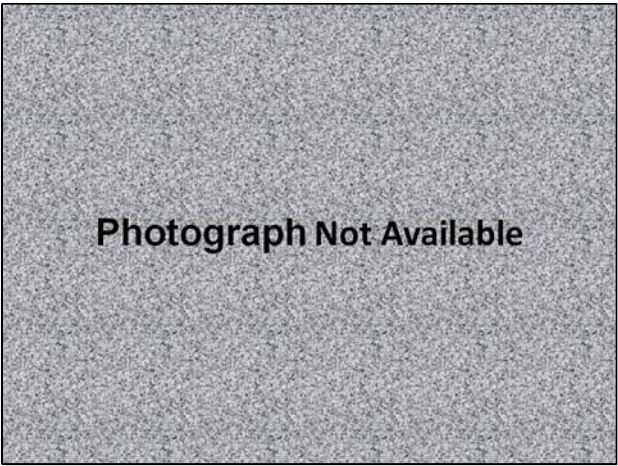
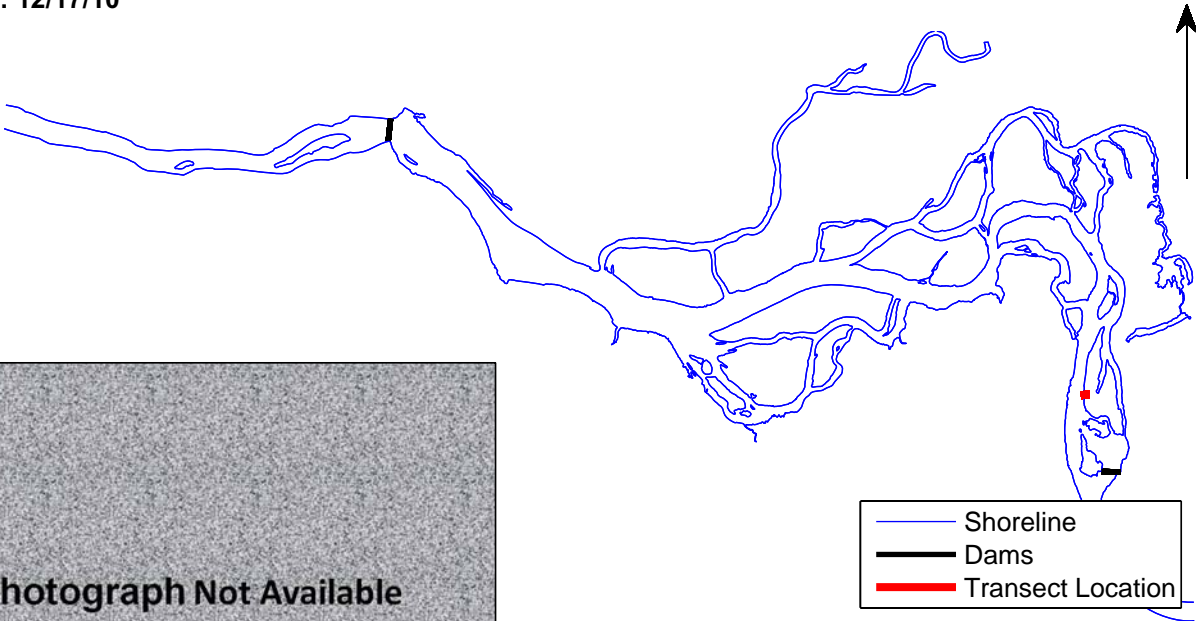
Otsego City Area 2 Bank Profiles
Transect: OCBN-15
Surveyed: 12/16/10

DRAFT



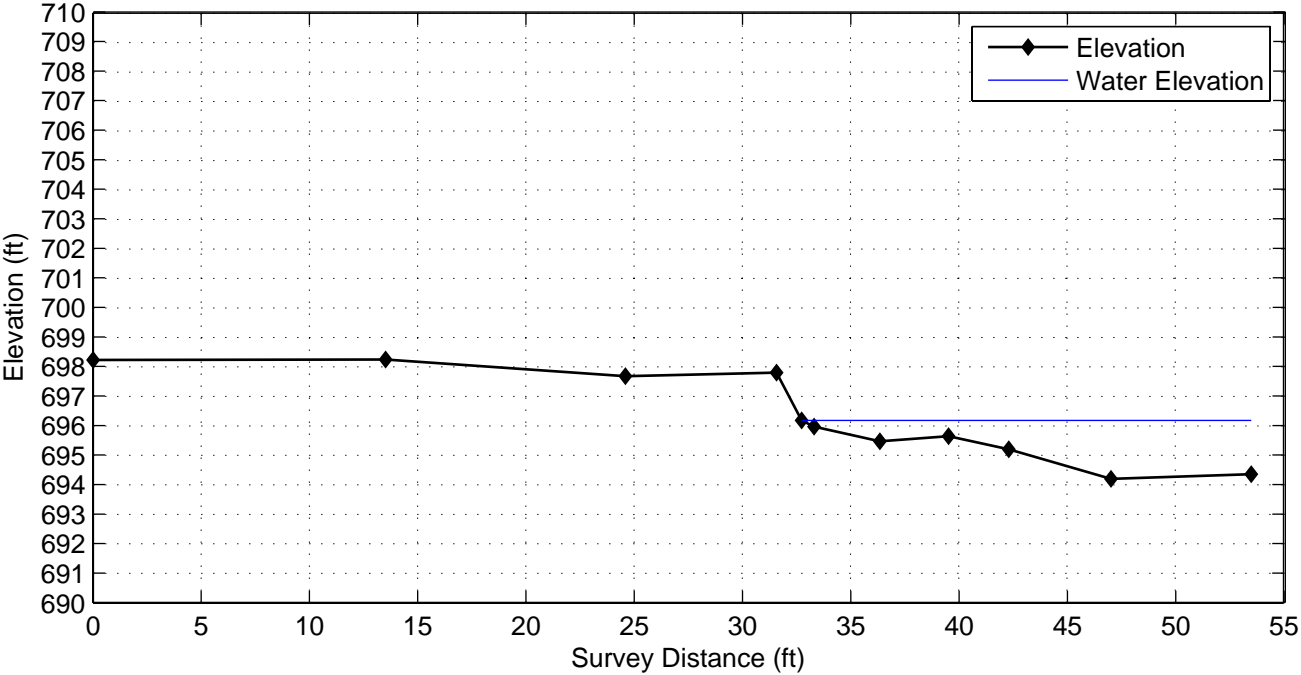
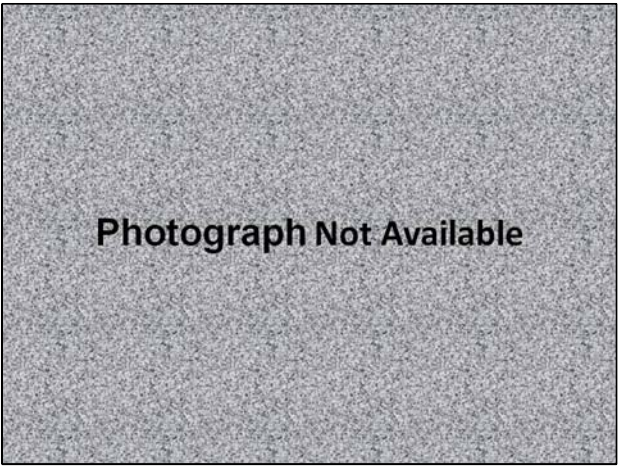
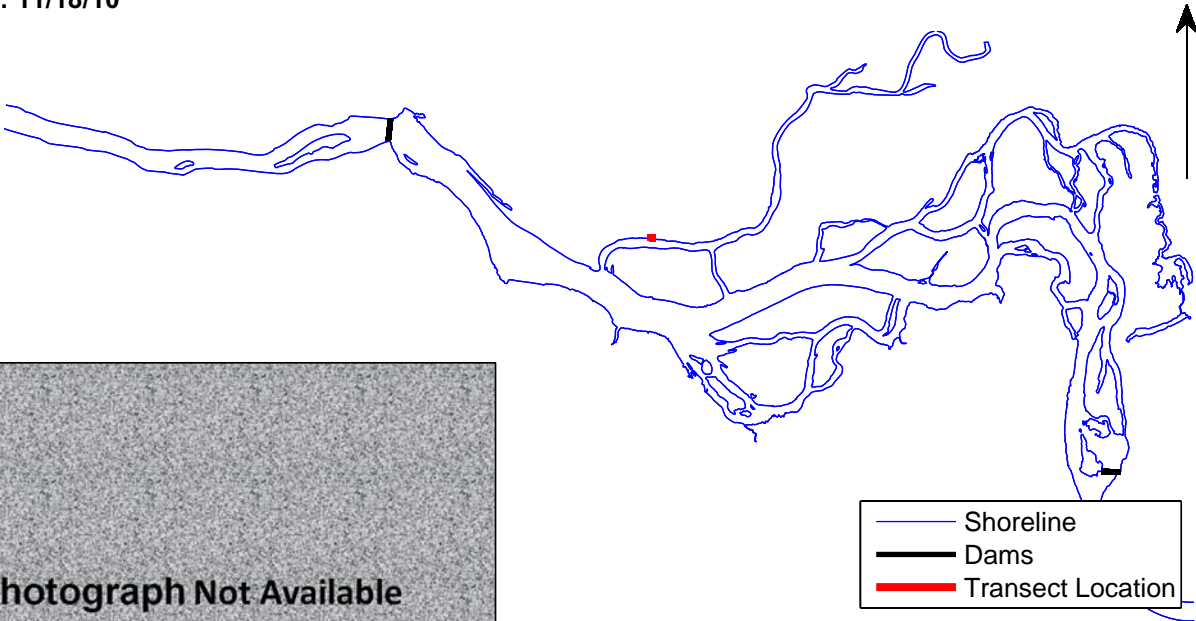
Otsego City Area 2 Bank Profiles
Transect: OCBN-16
Surveyed: 12/17/10

DRAFT



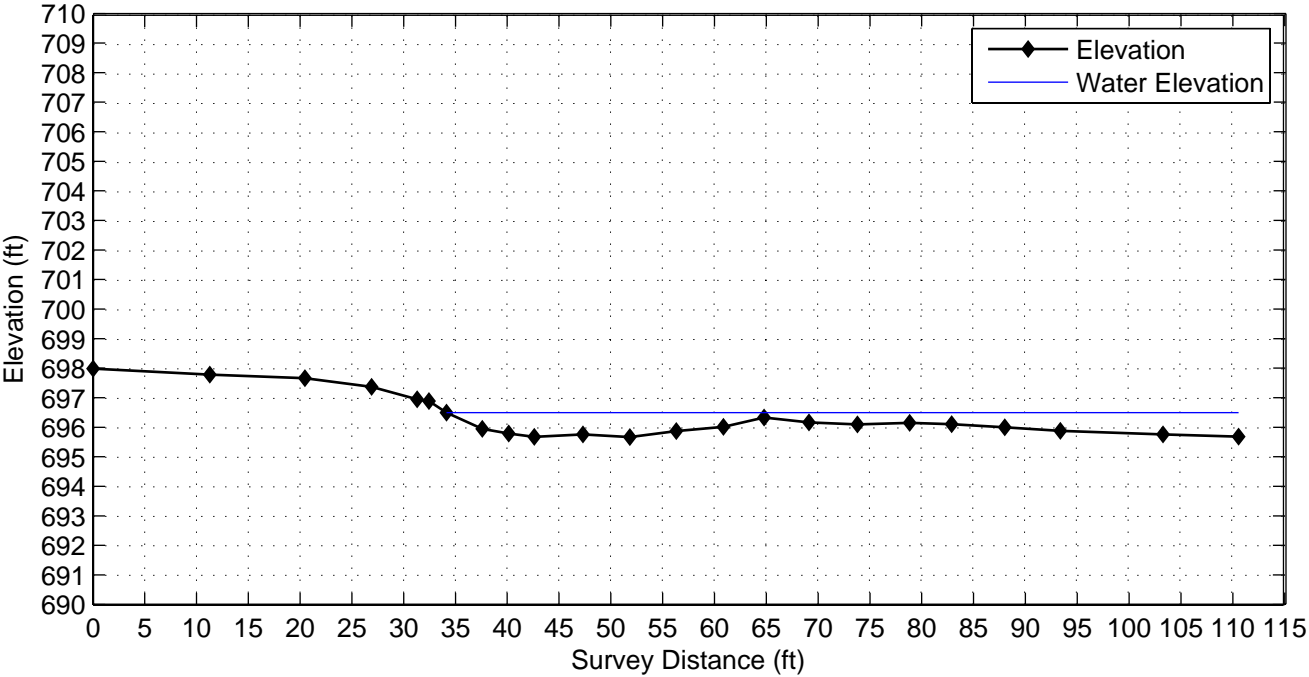
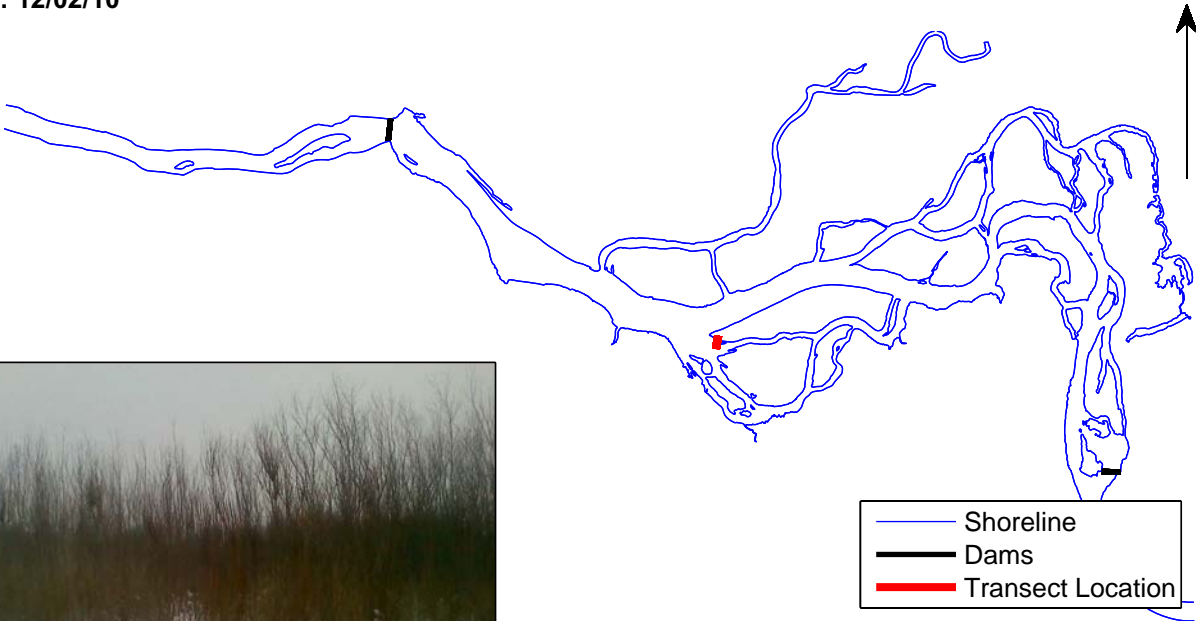
Otsego City Area 2 Bank Profiles
Transect: OCBN-17
Surveyed: 11/18/10

DRAFT



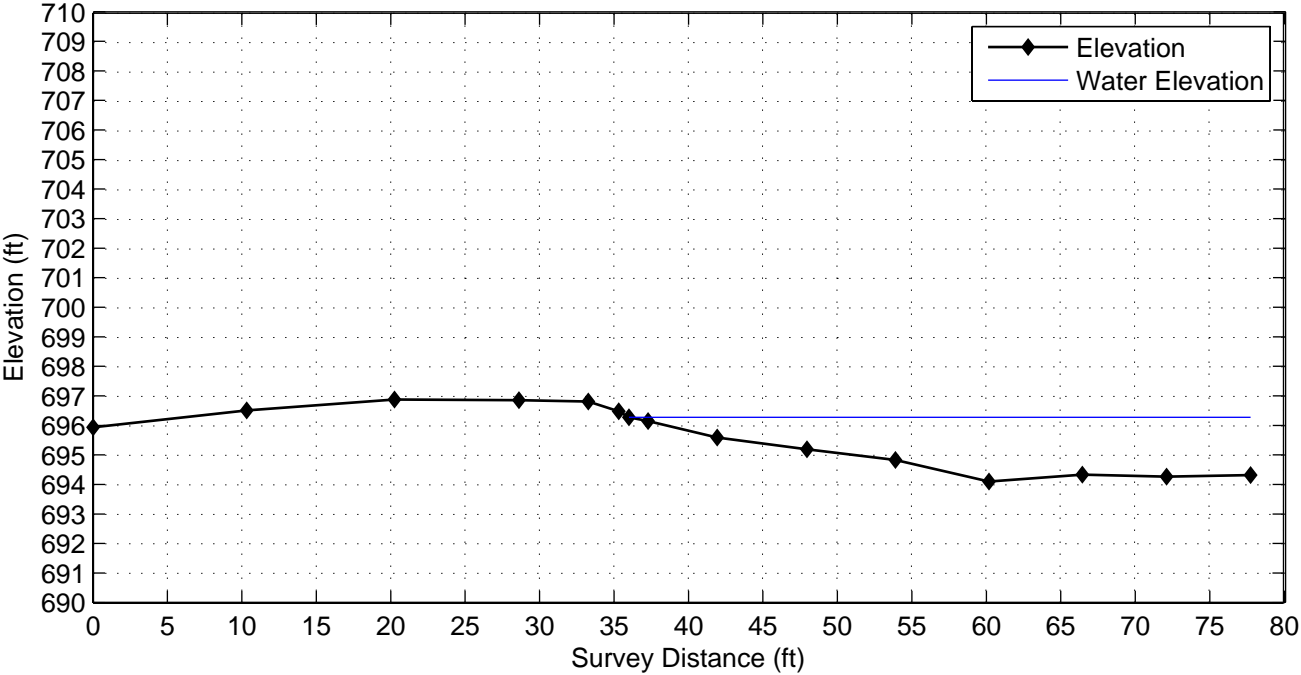
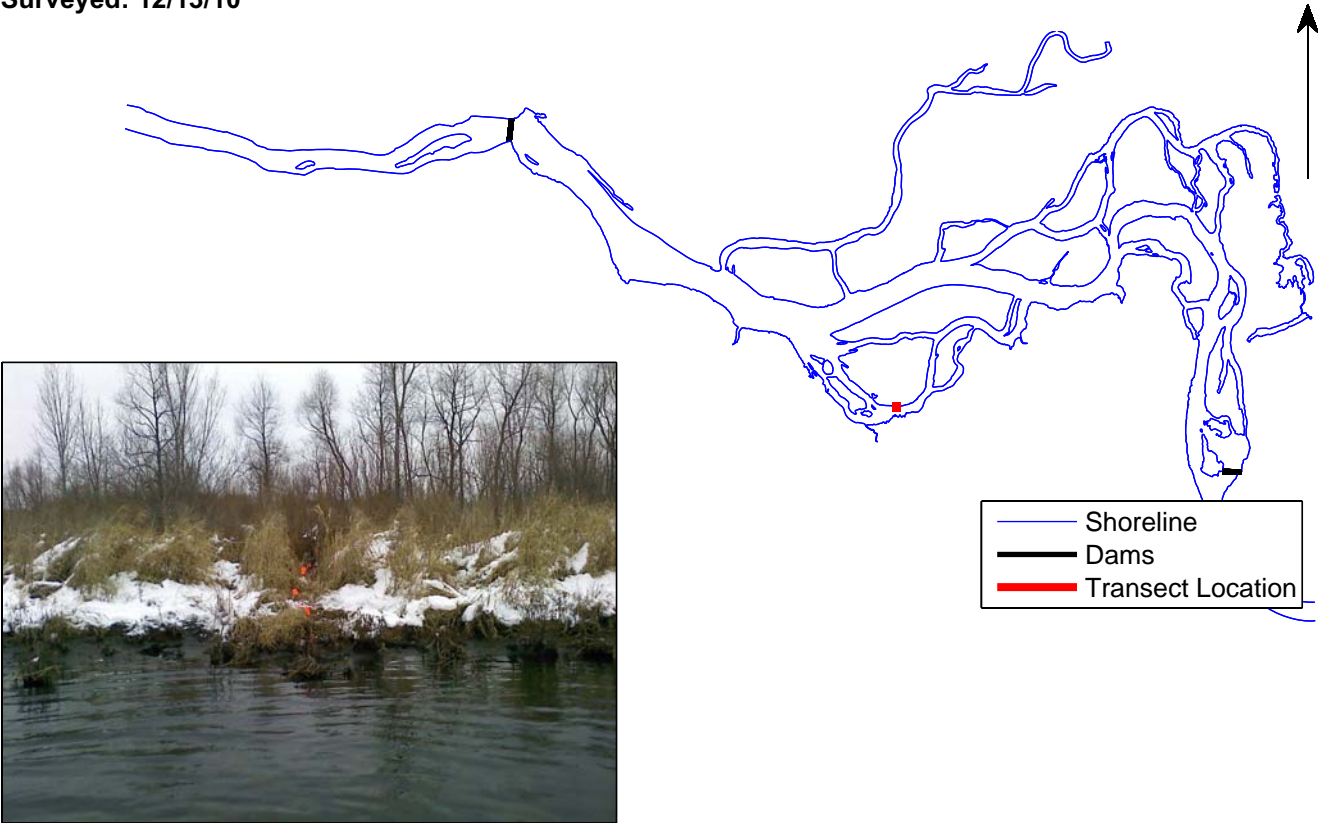
Otsego City Area 2 Bank Profiles
Transect: OCBN-18
Surveyed: 12/02/10

DRAFT



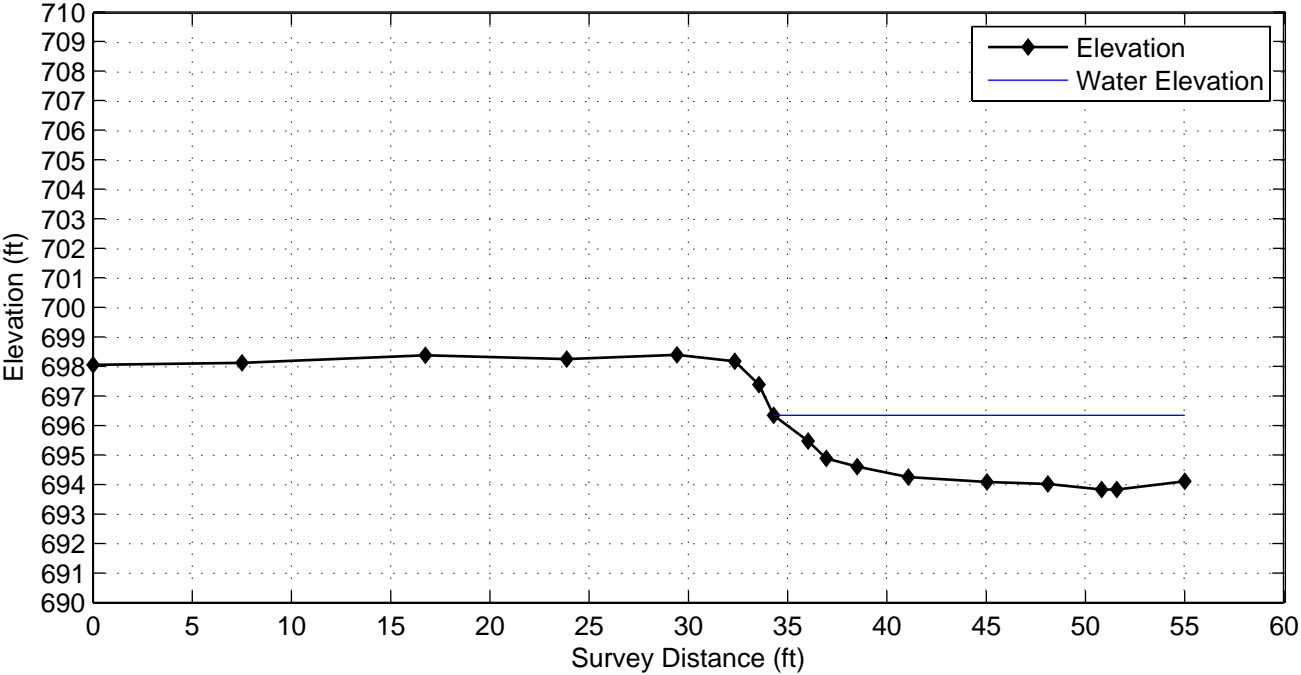
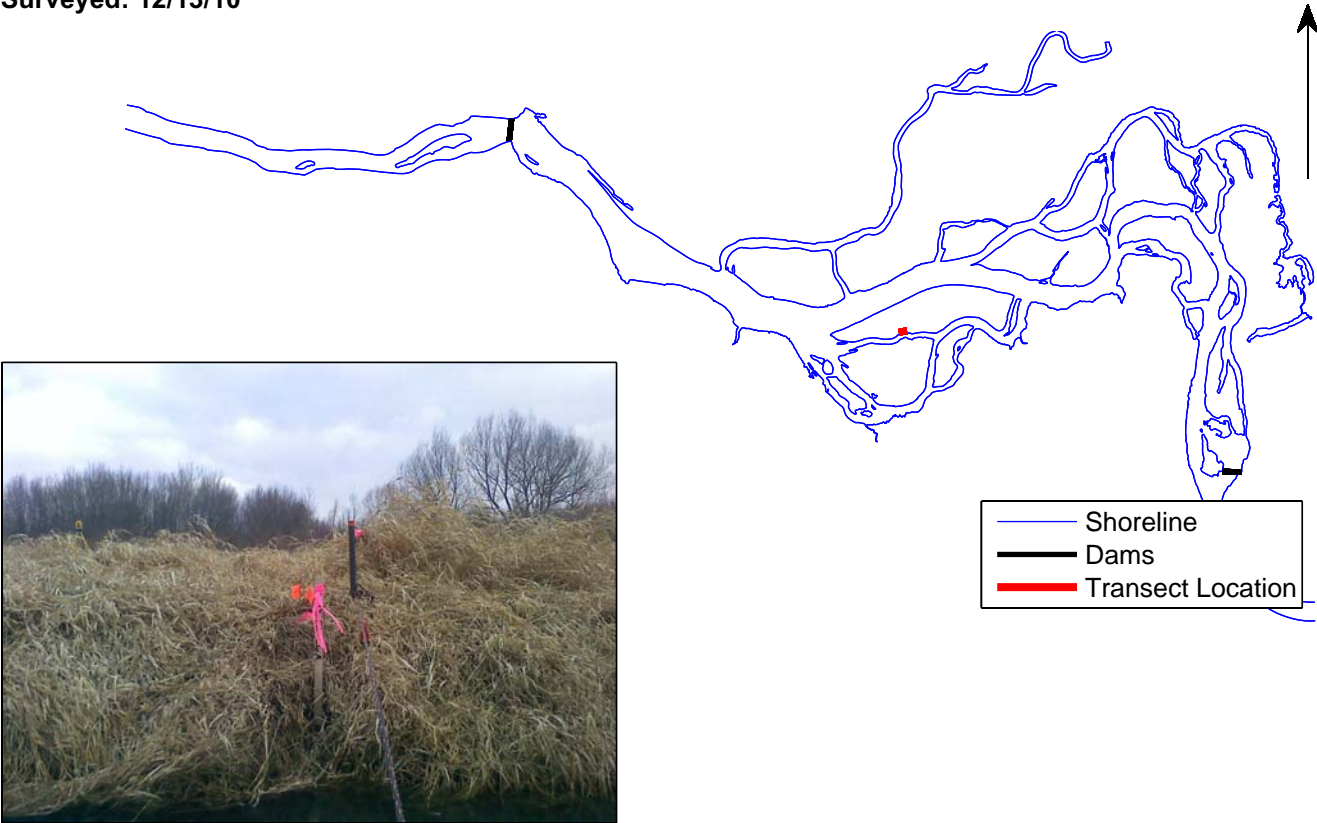
Otsego City Area 2 Bank Profiles
Transect: OCBN-19
Surveyed: 12/13/10

DRAFT



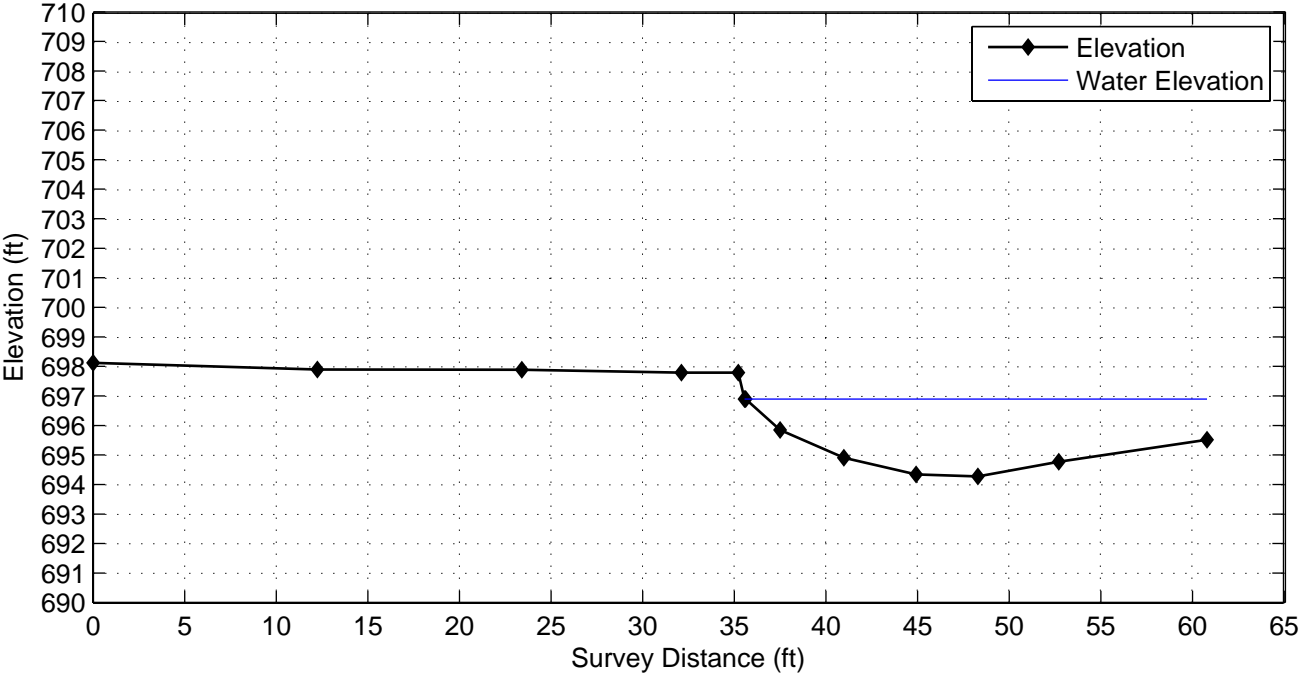
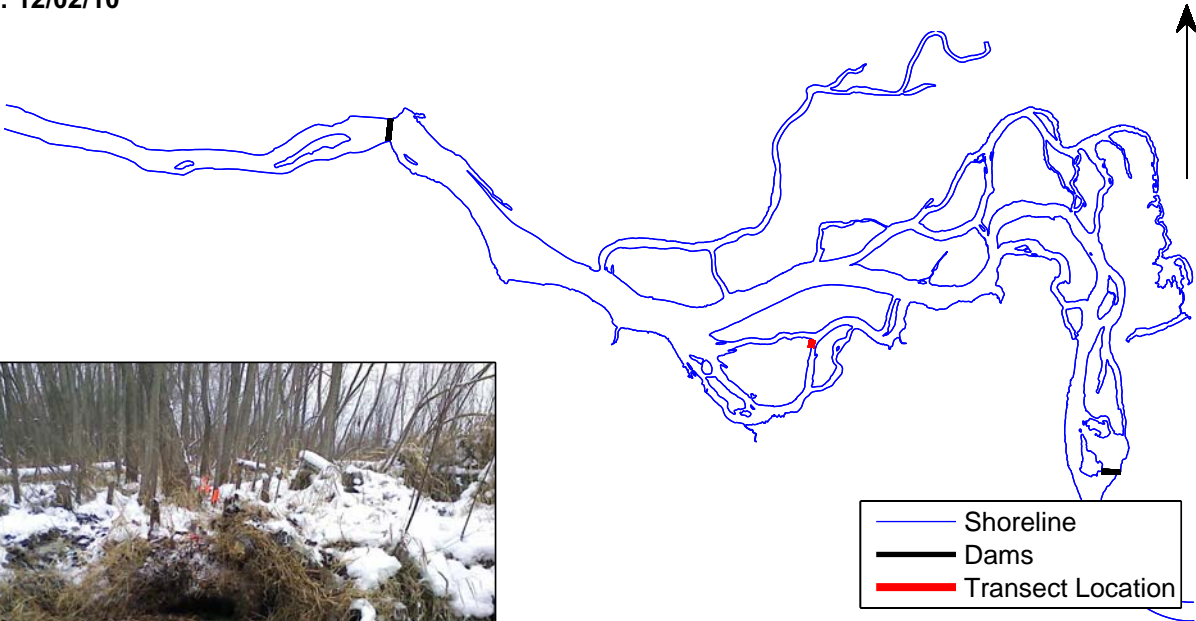
Otsego City Area 2 Bank Profiles
 Transect: OCBN-20
 Surveyed: 12/13/10

DRAFT



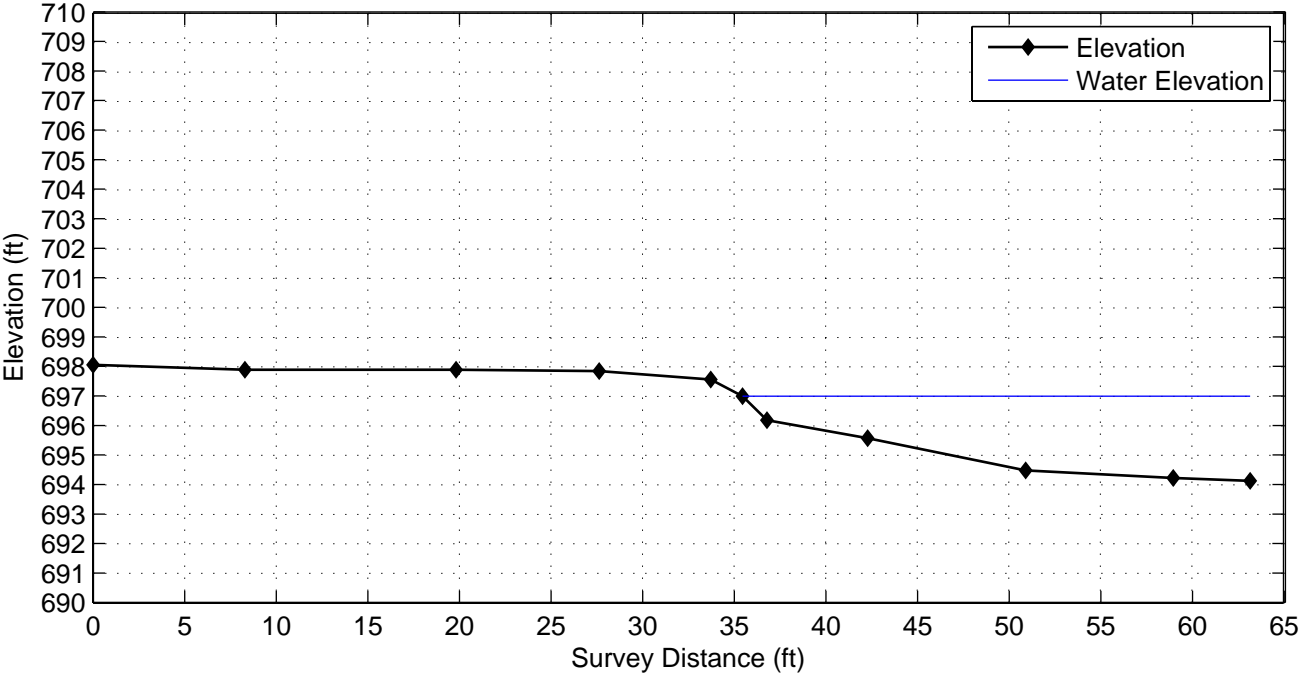
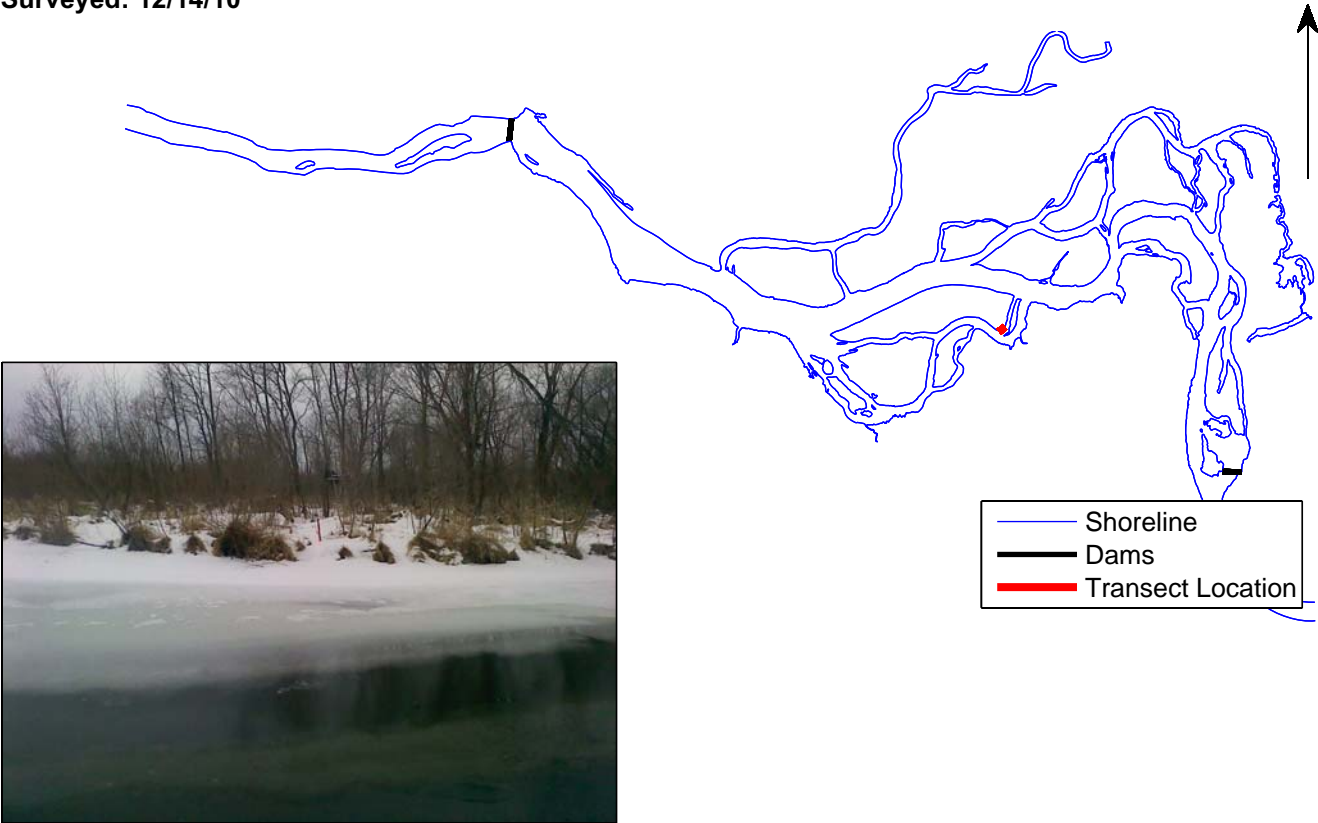
Otsego City Area 2 Bank Profiles
Transect: OCBN-21
Surveyed: 12/02/10

DRAFT



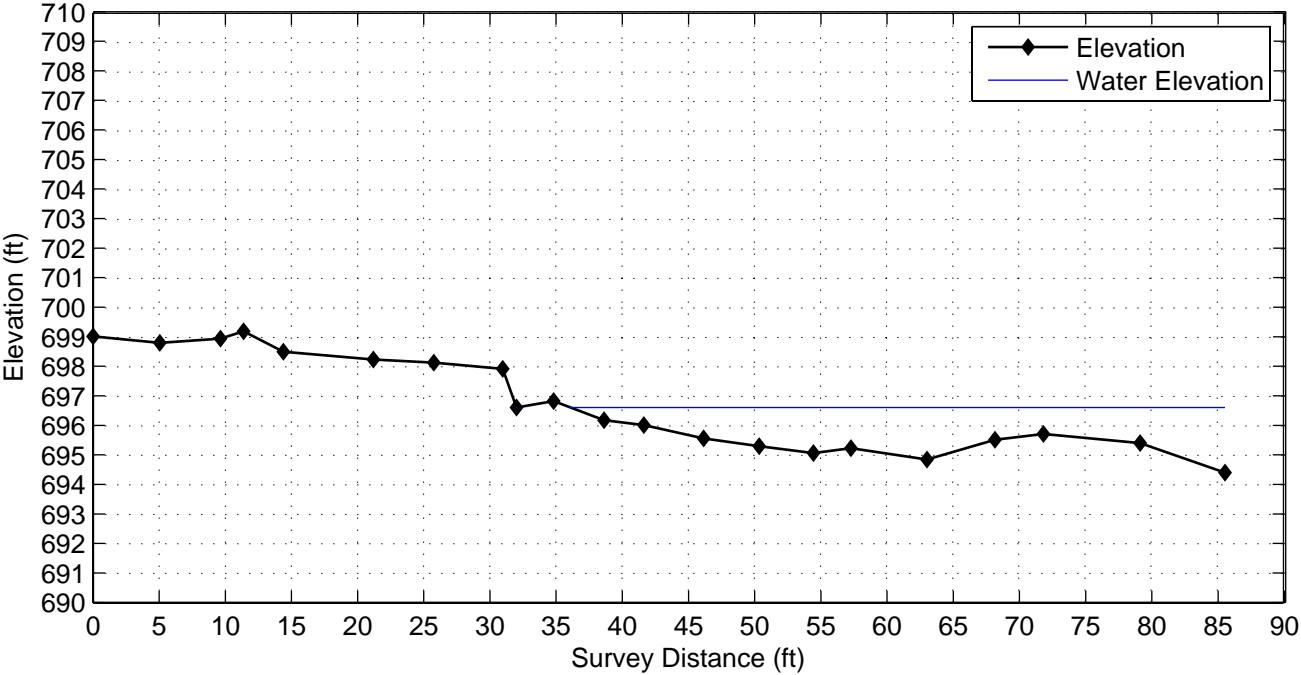
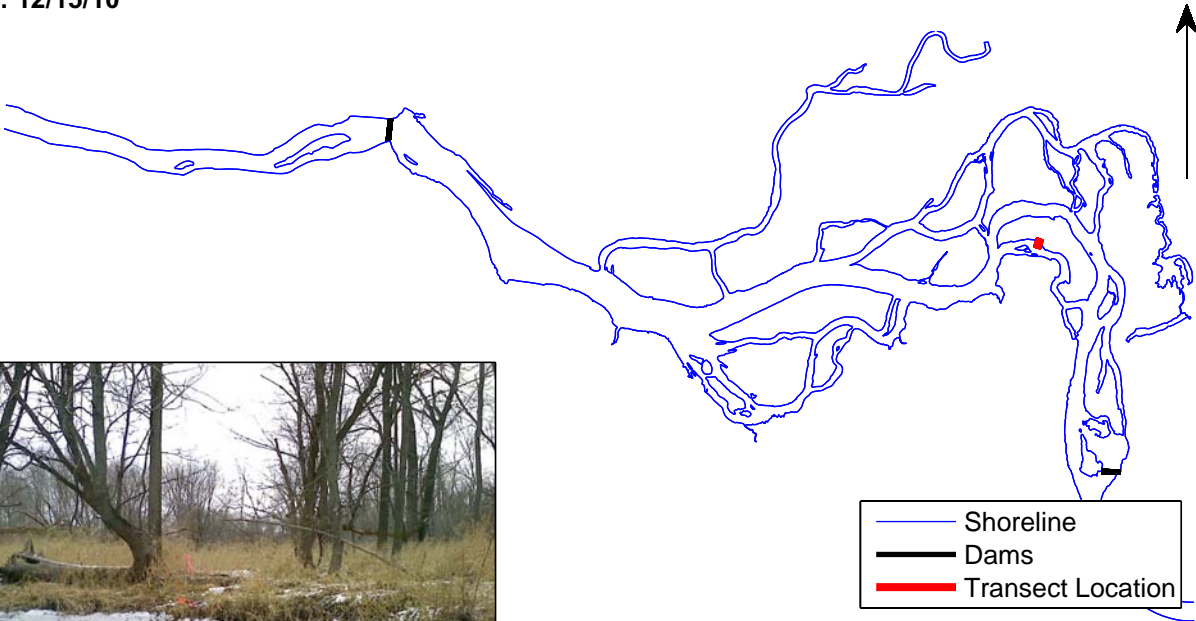
Otsego City Area 2 Bank Profiles
Transect: OCBN-22
Surveyed: 12/14/10

DRAFT



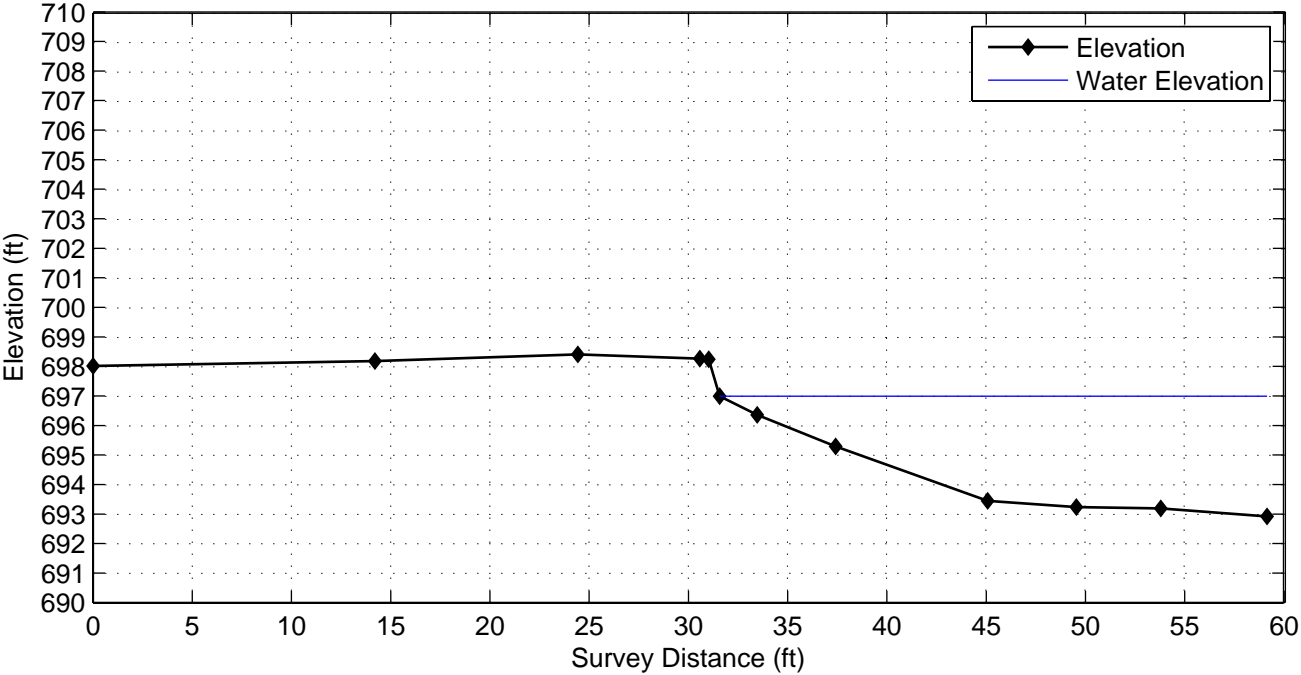
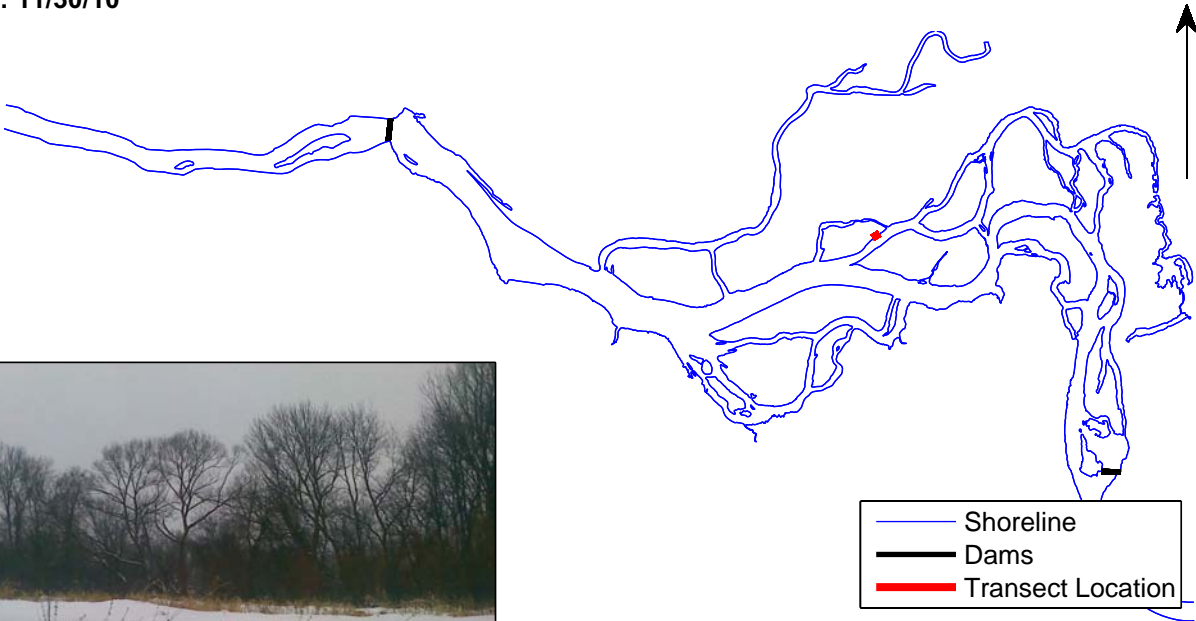
Otsego City Area 2 Bank Profiles
Transect: OCBN-23
Surveyed: 12/15/10

DRAFT



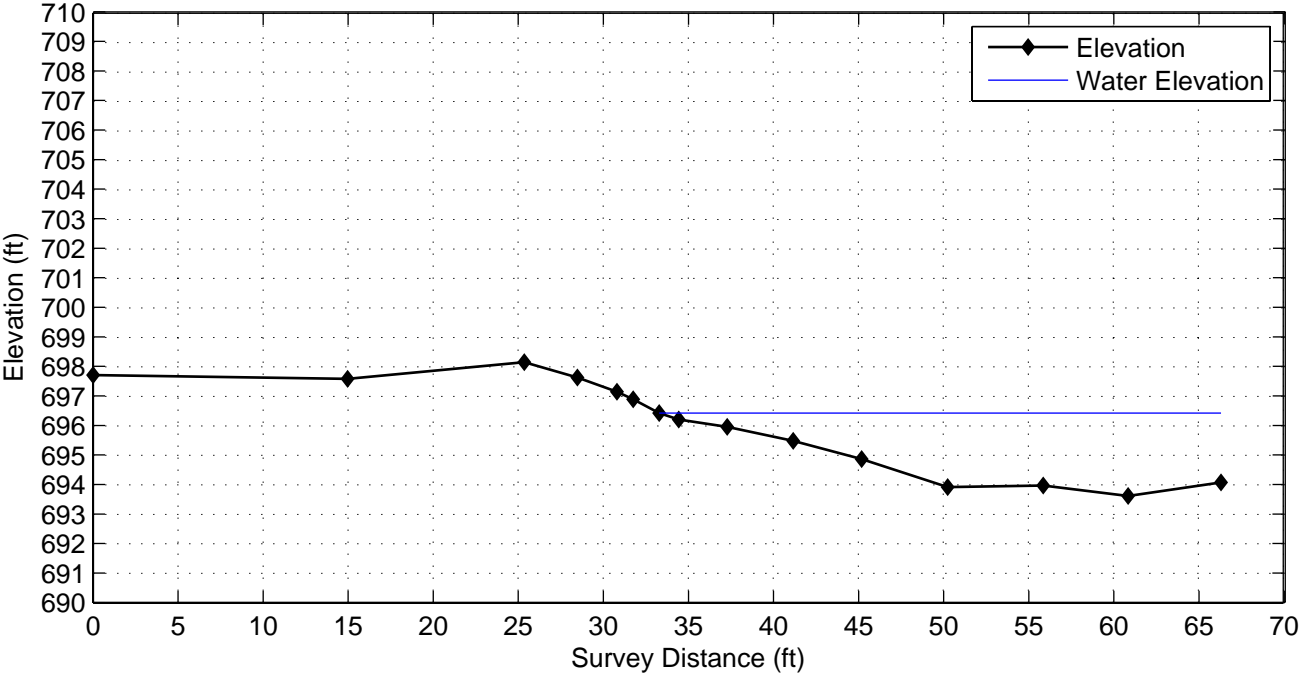
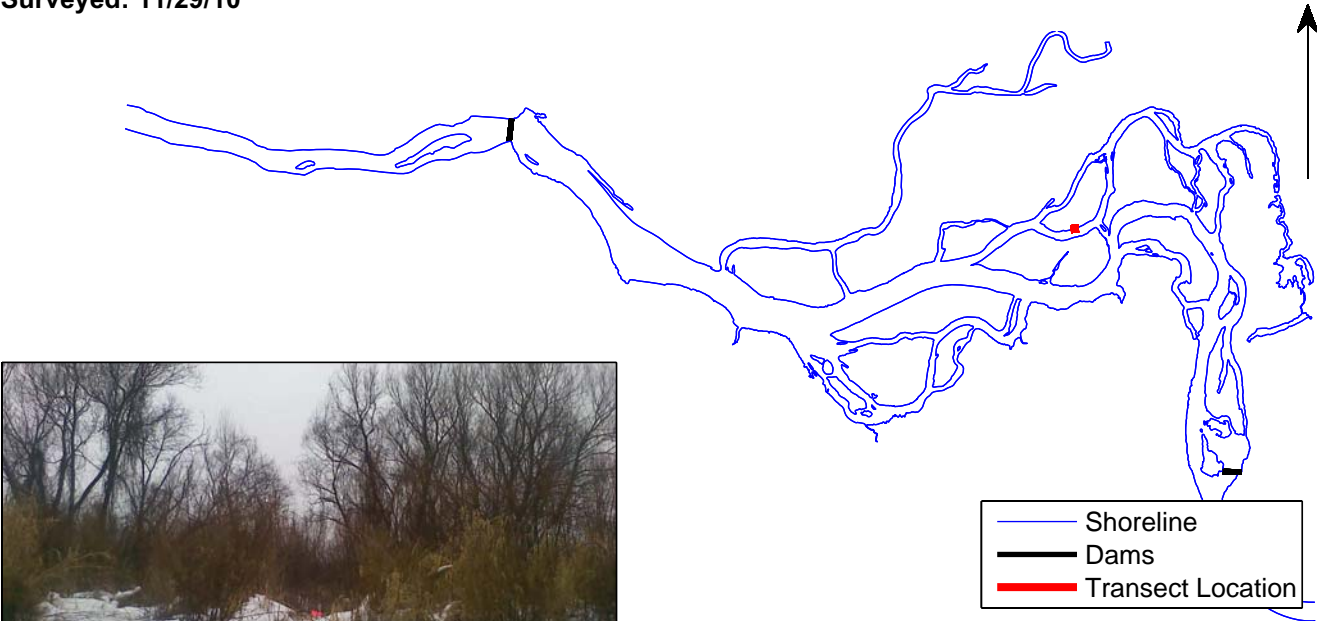
Otsego City Area 2 Bank Profiles
Transect: OCBN-24
Surveyed: 11/30/10

DRAFT



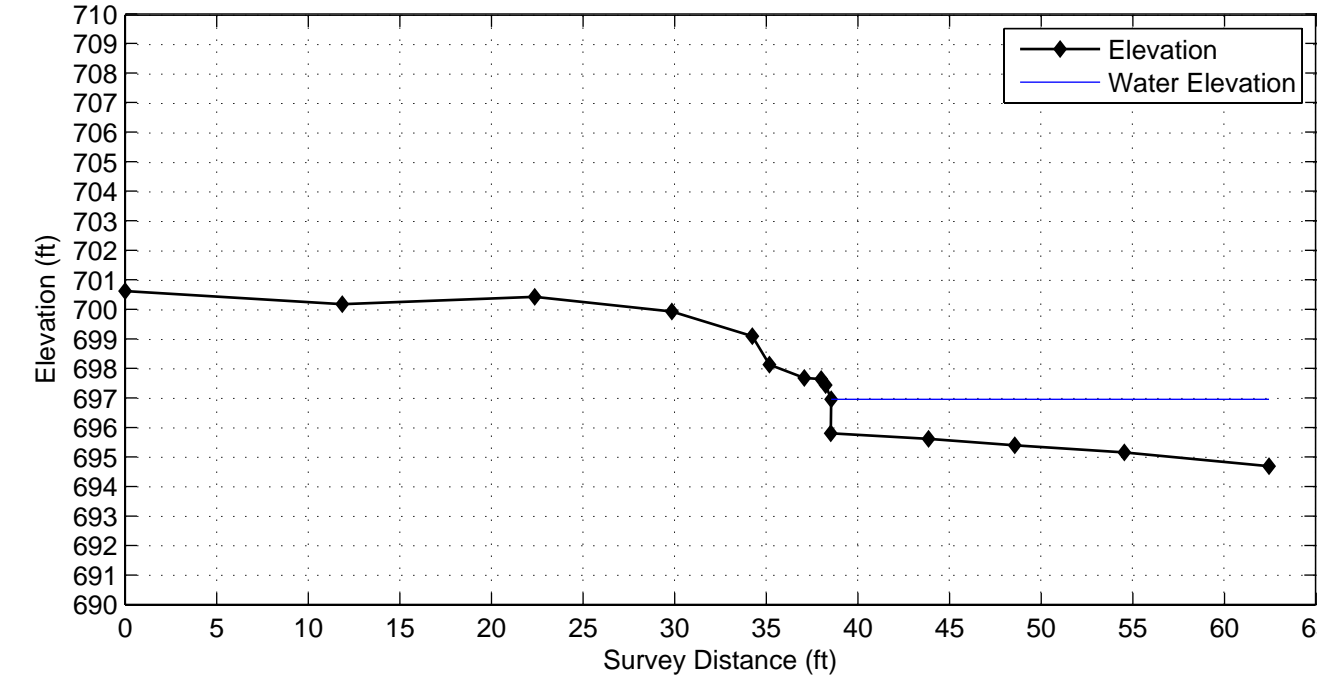
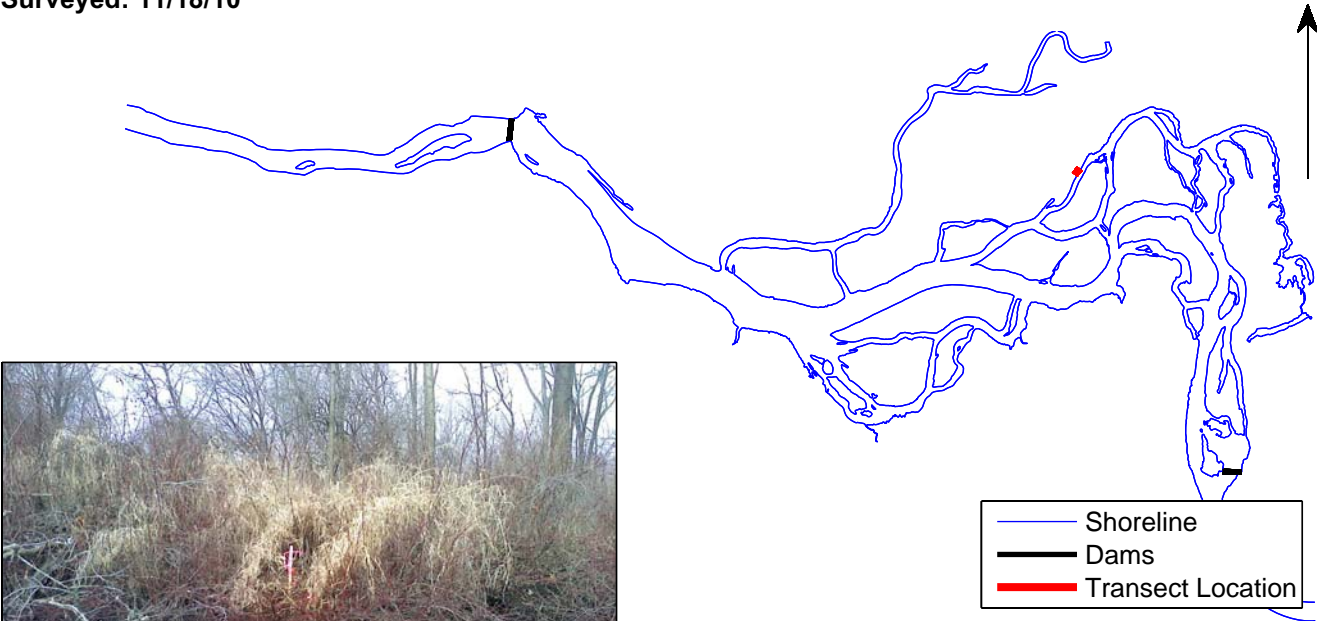
Otsego City Area 2 Bank Profiles
Transect: OCBN-25
Surveyed: 11/29/10

DRAFT



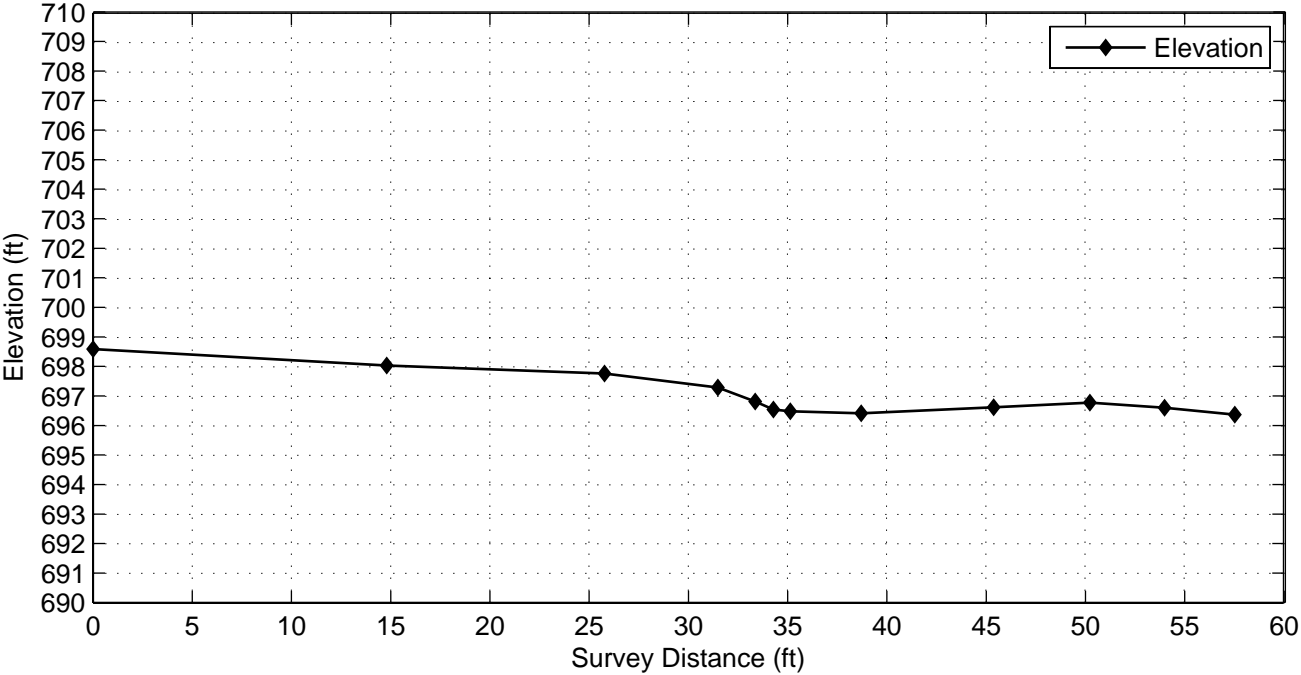
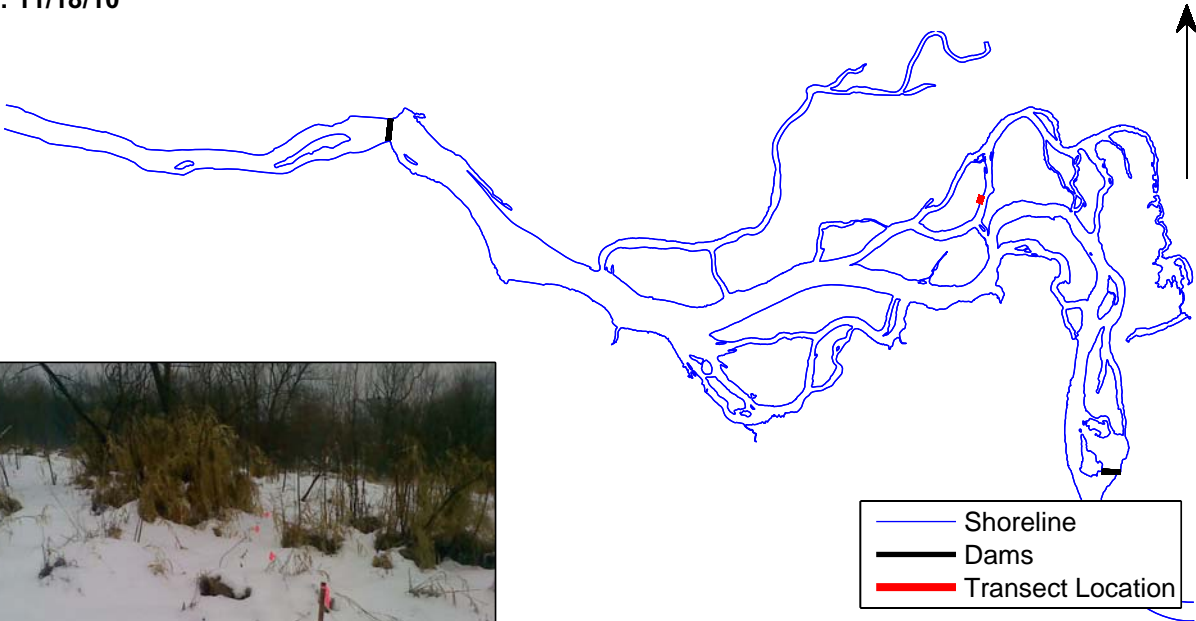
Otsego City Area 2 Bank Profiles
Transect: OCBN-26
Surveyed: 11/18/10

DRAFT



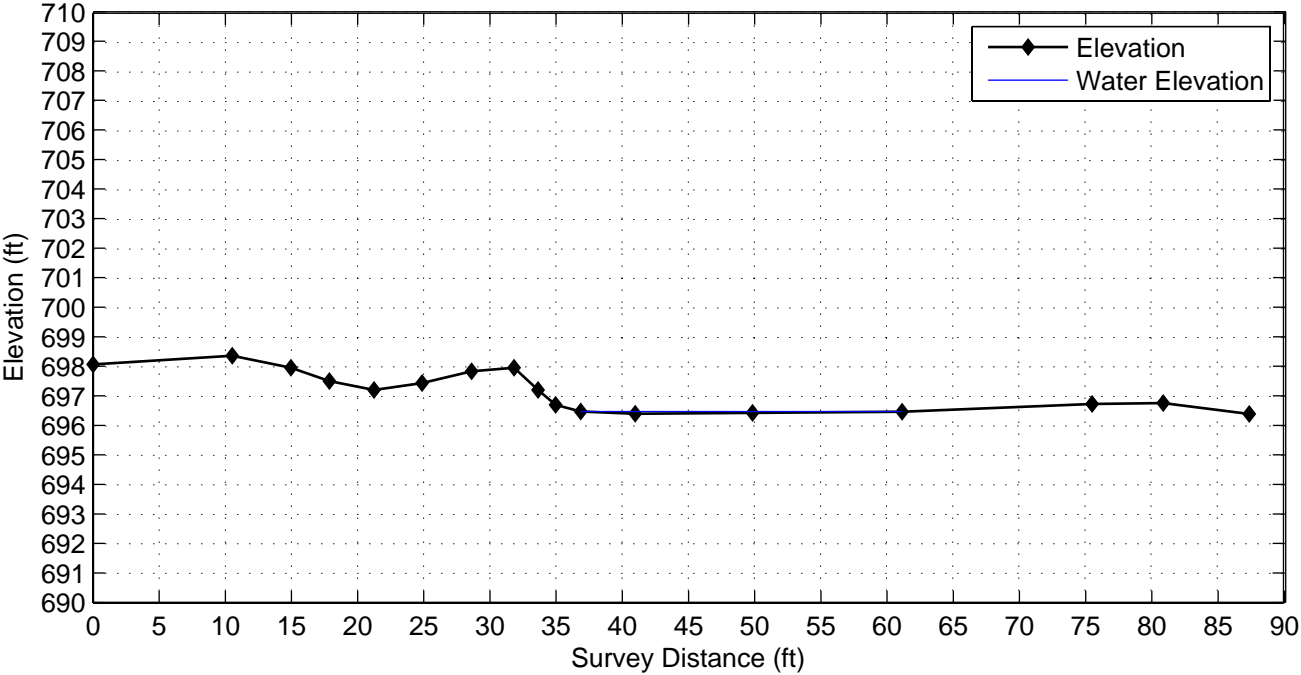
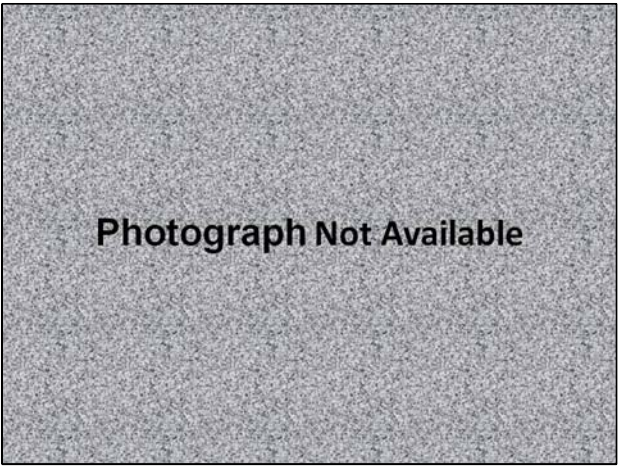
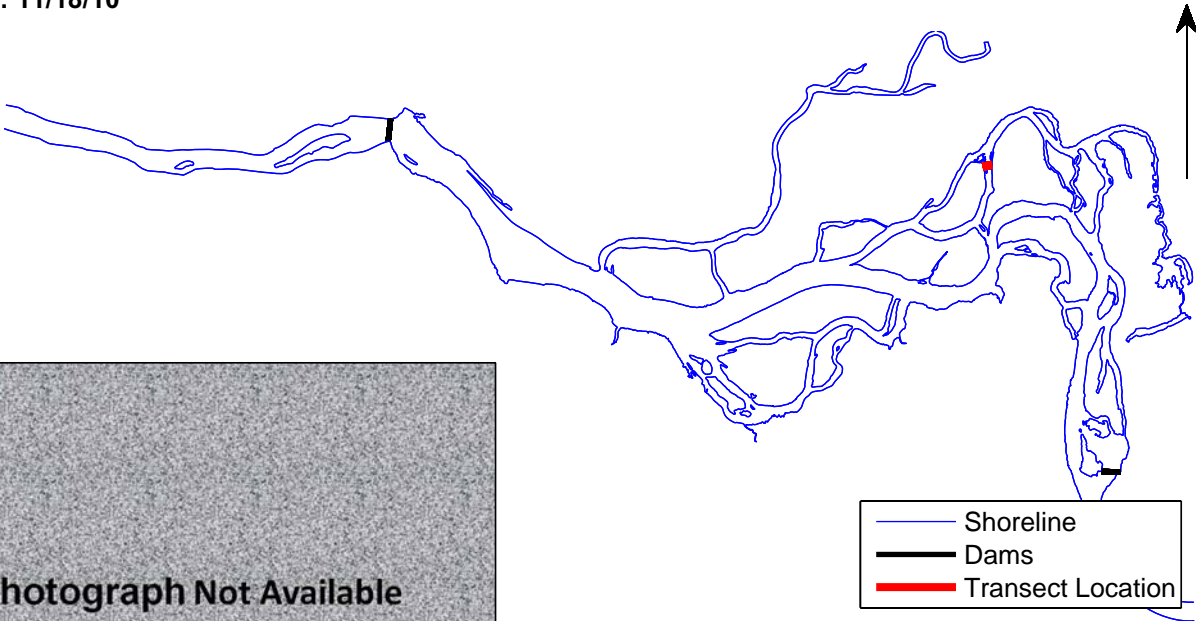
Otsego City Area 2 Bank Profiles
Transect: OCBN-27
Surveyed: 11/18/10

DRAFT



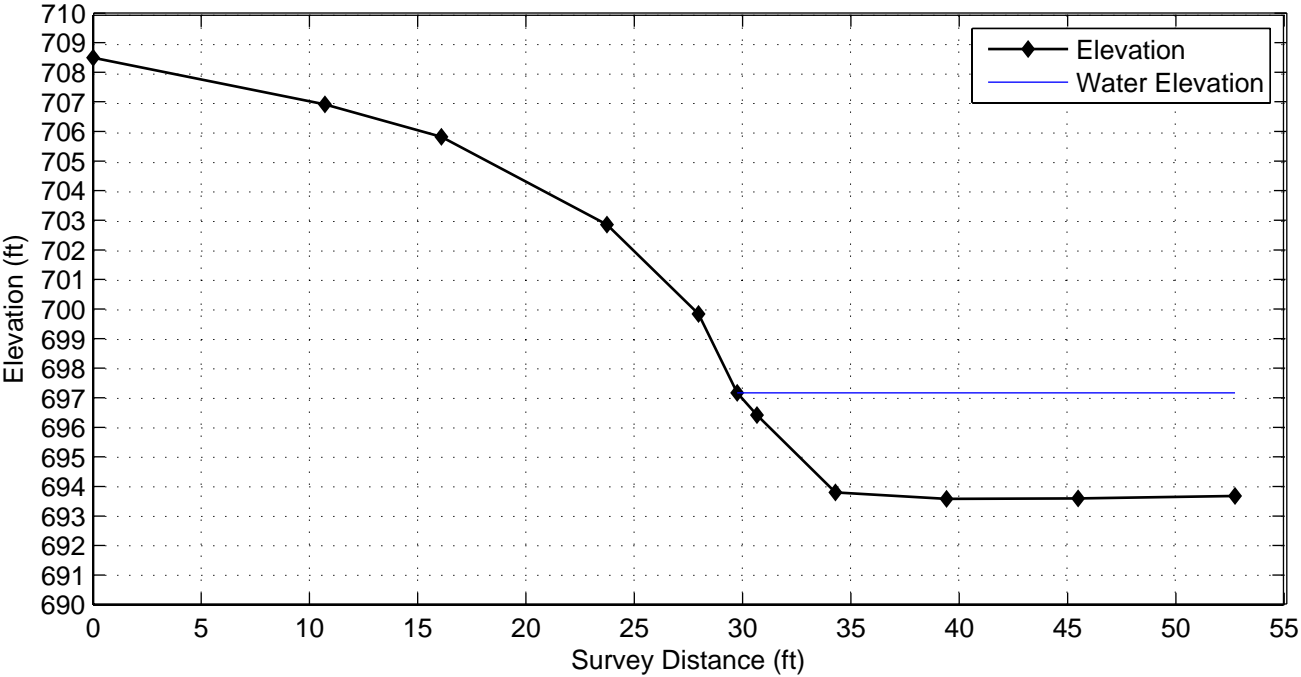
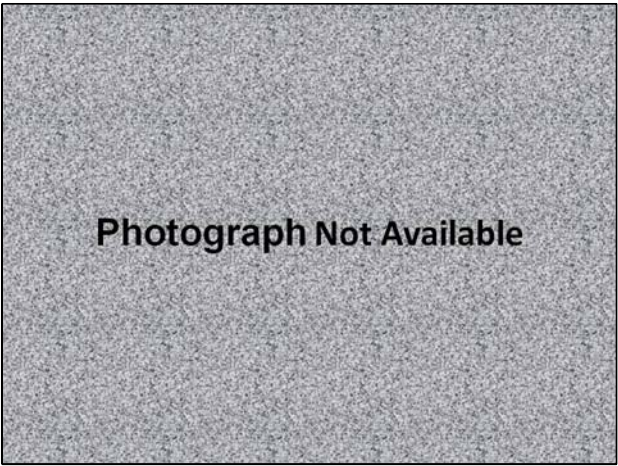
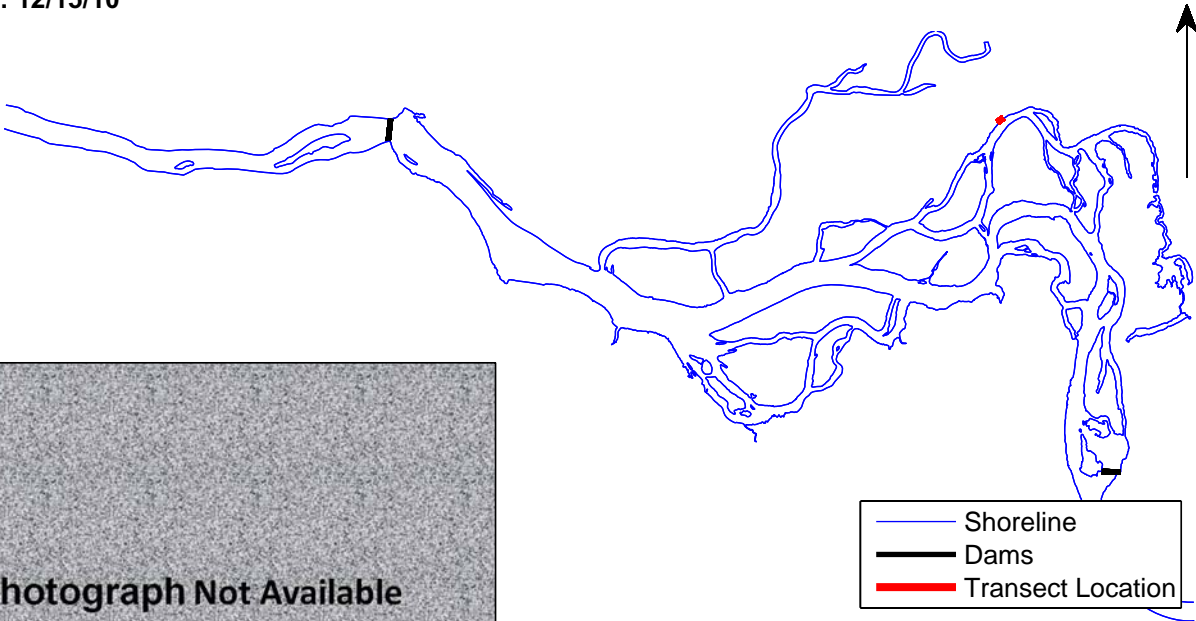
Otsego City Area 2 Bank Profiles
Transect: OCBN-28
Surveyed: 11/18/10

DRAFT



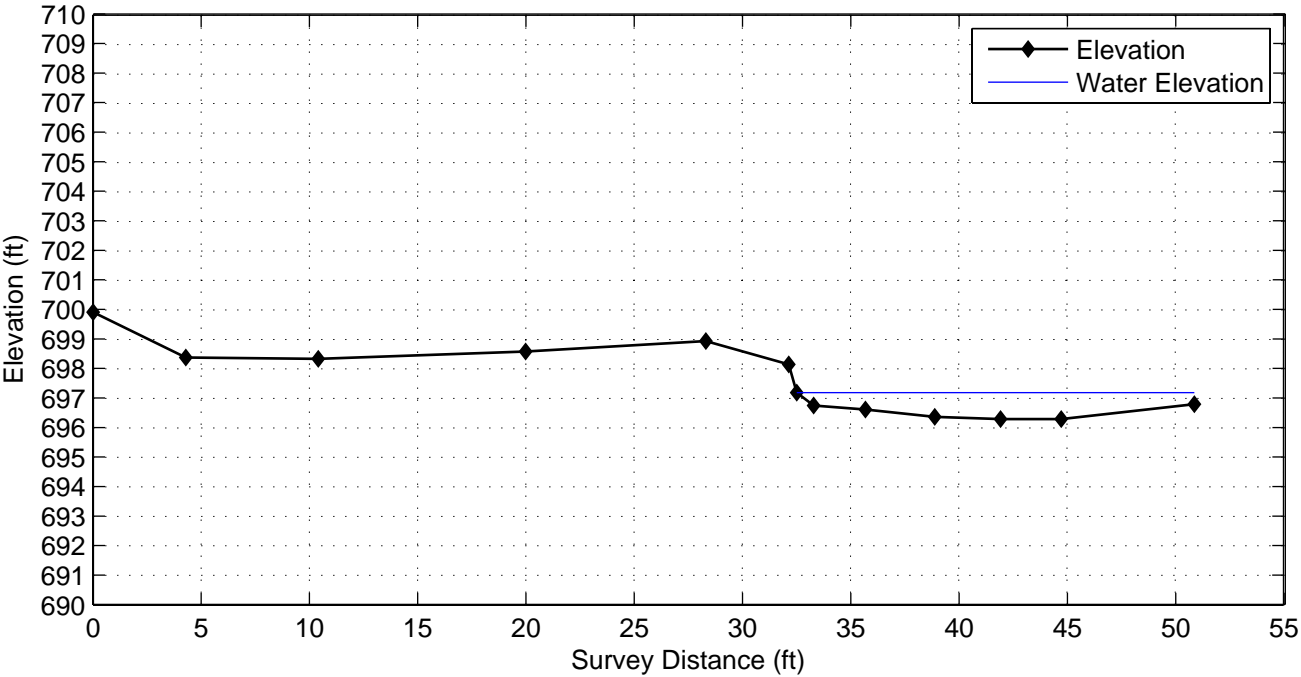
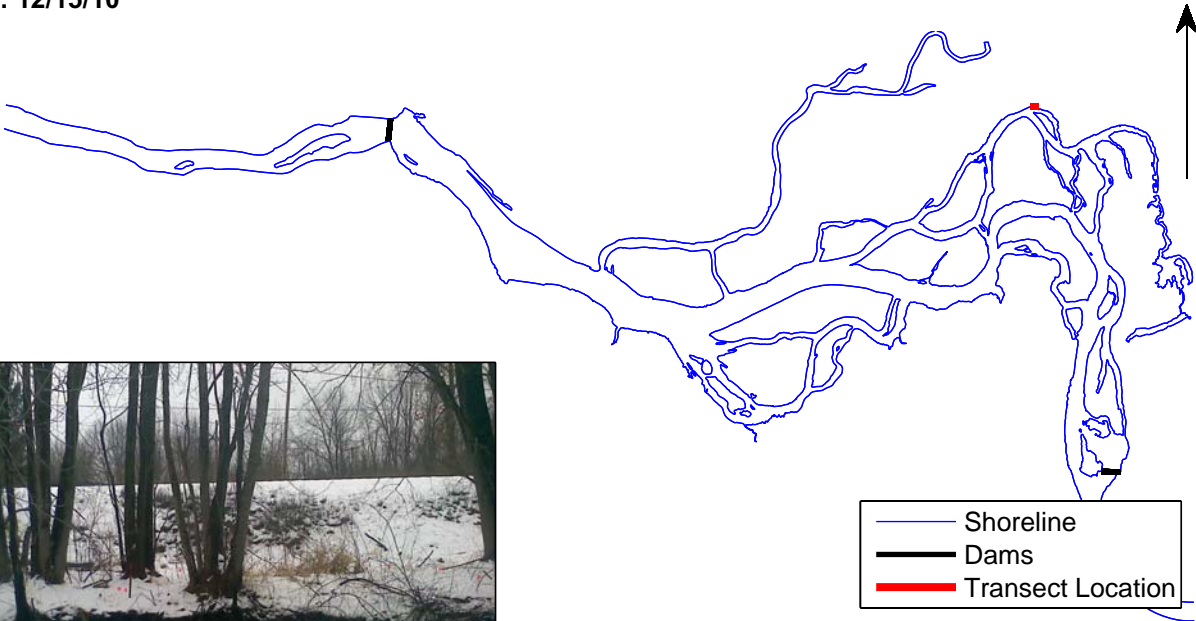
Otsego City Area 2 Bank Profiles
Transect: OCBN-29
Surveyed: 12/15/10

DRAFT



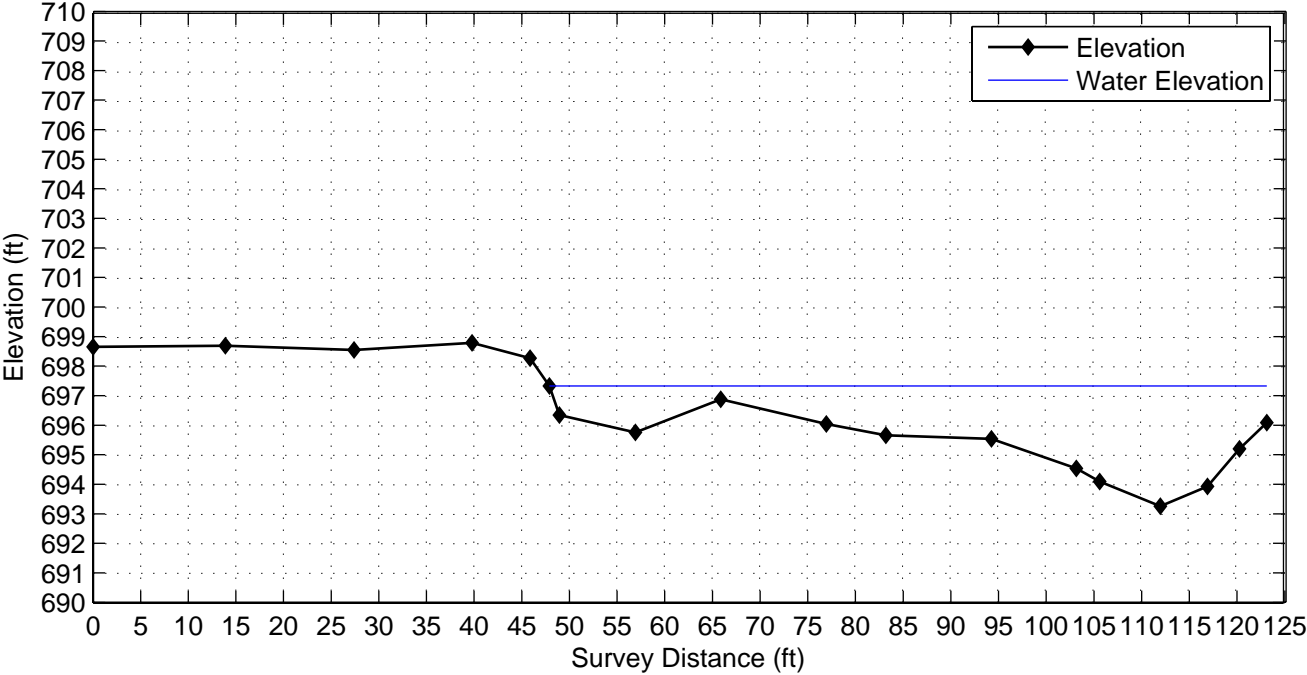
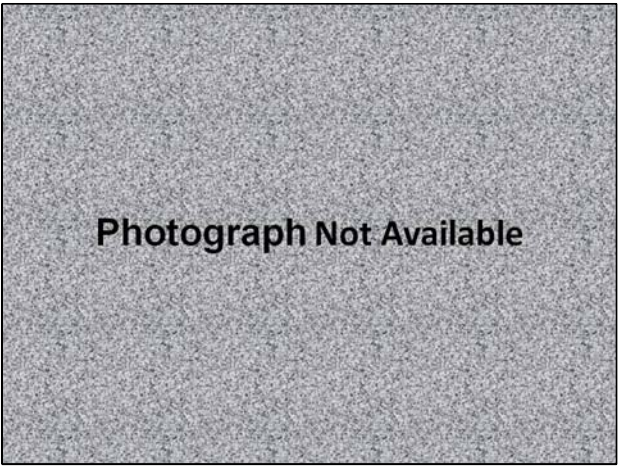
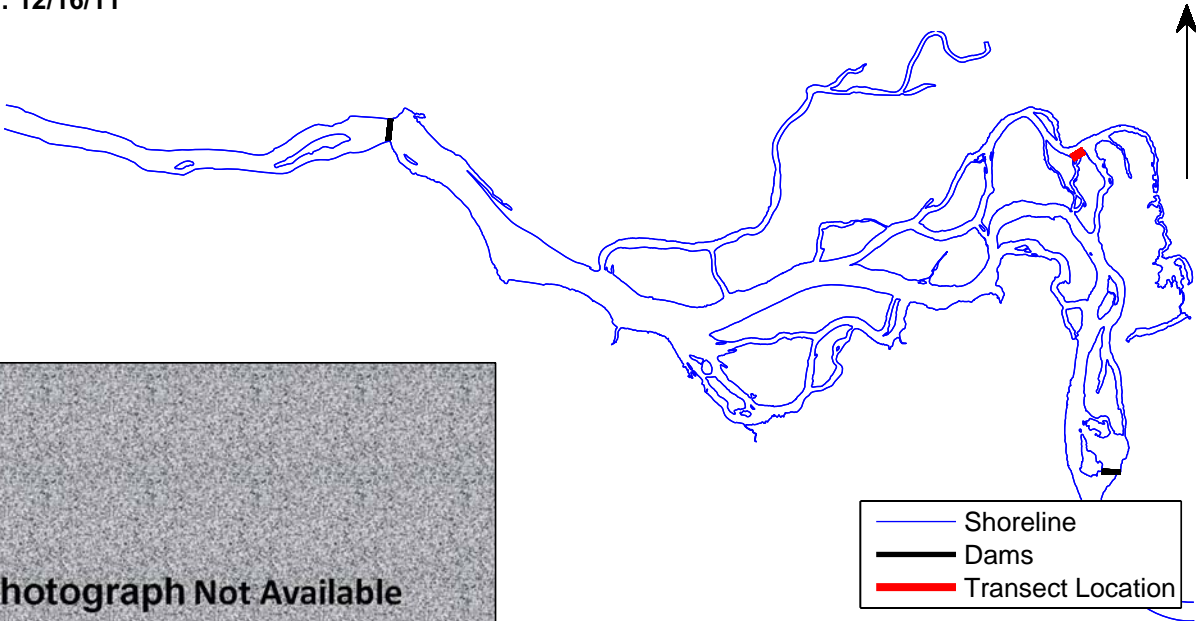
Otsego City Area 2 Bank Profiles
Transect: OCBN-30
Surveyed: 12/15/10

DRAFT



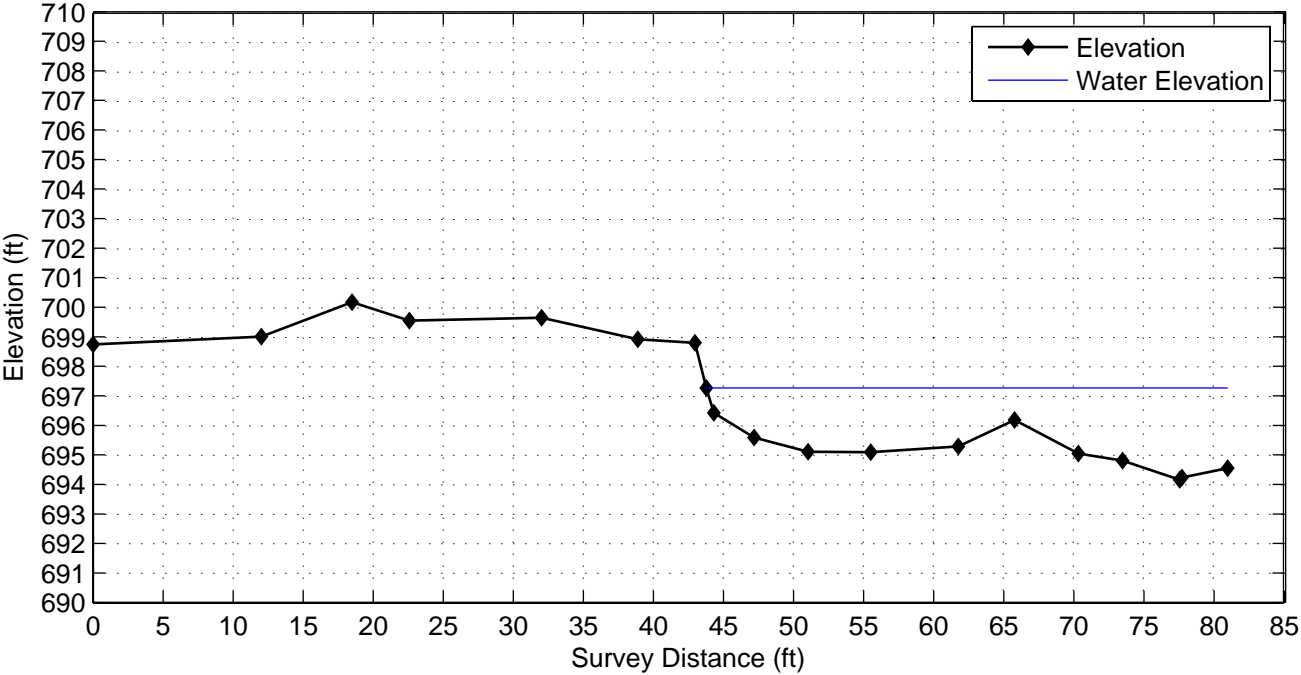
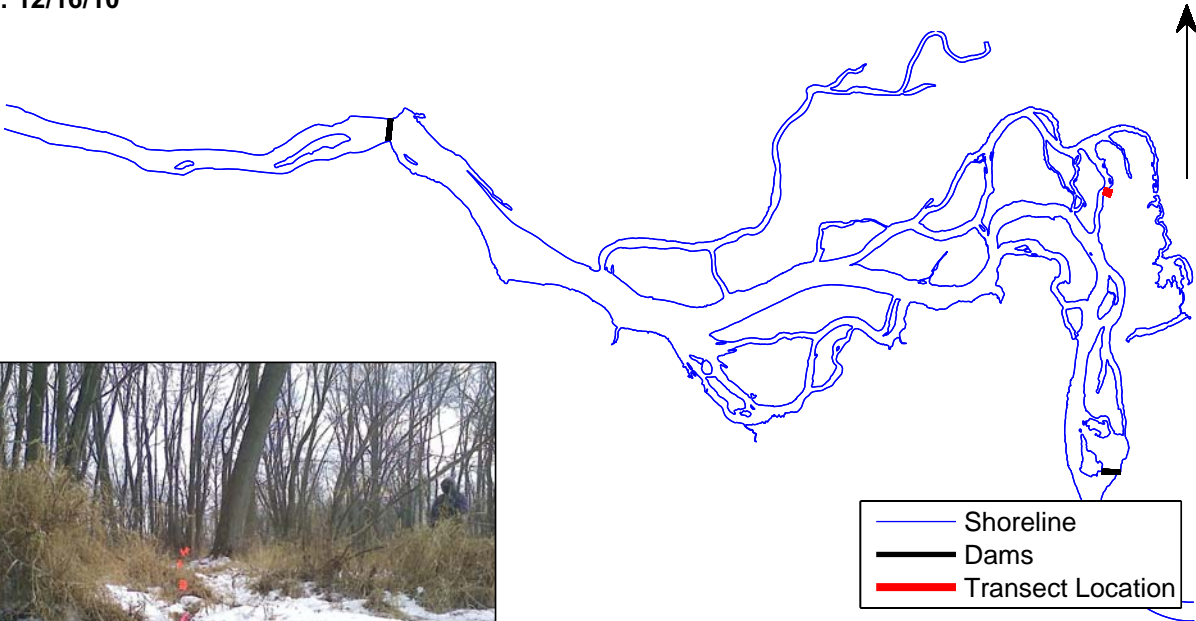
Otsego City Area 2 Bank Profiles
Transect: OCBN-31
Surveyed: 12/16/11

DRAFT



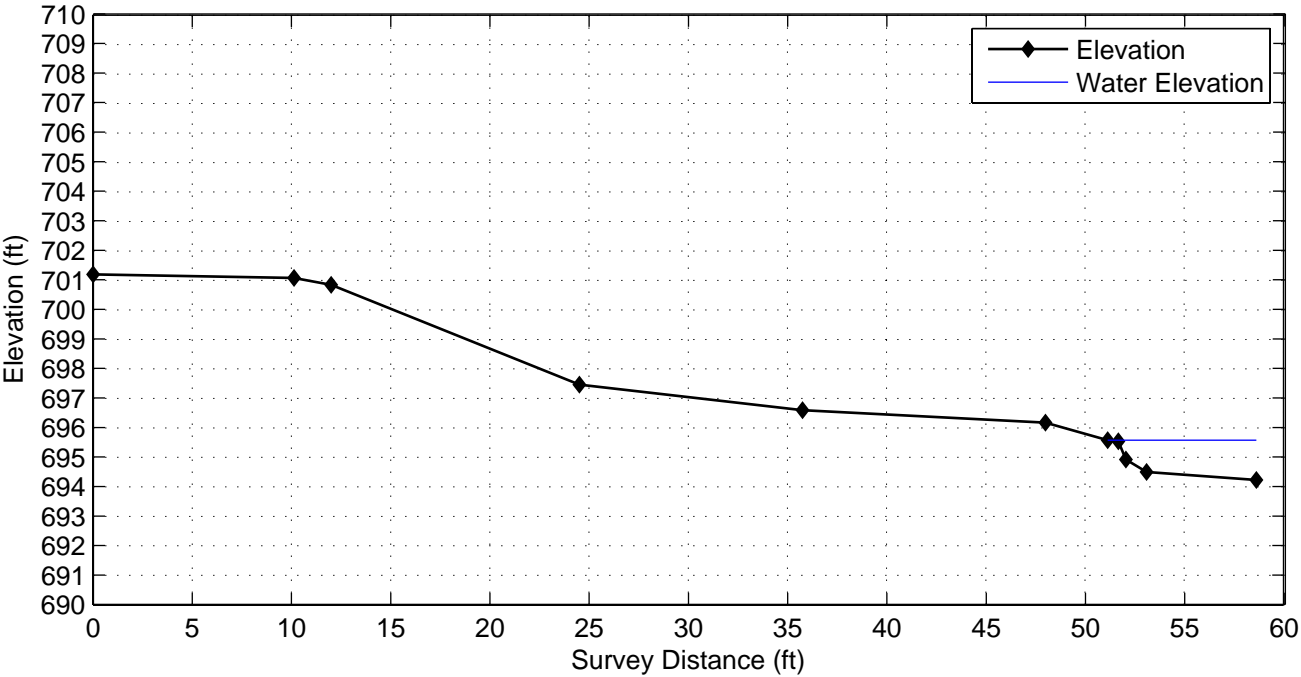
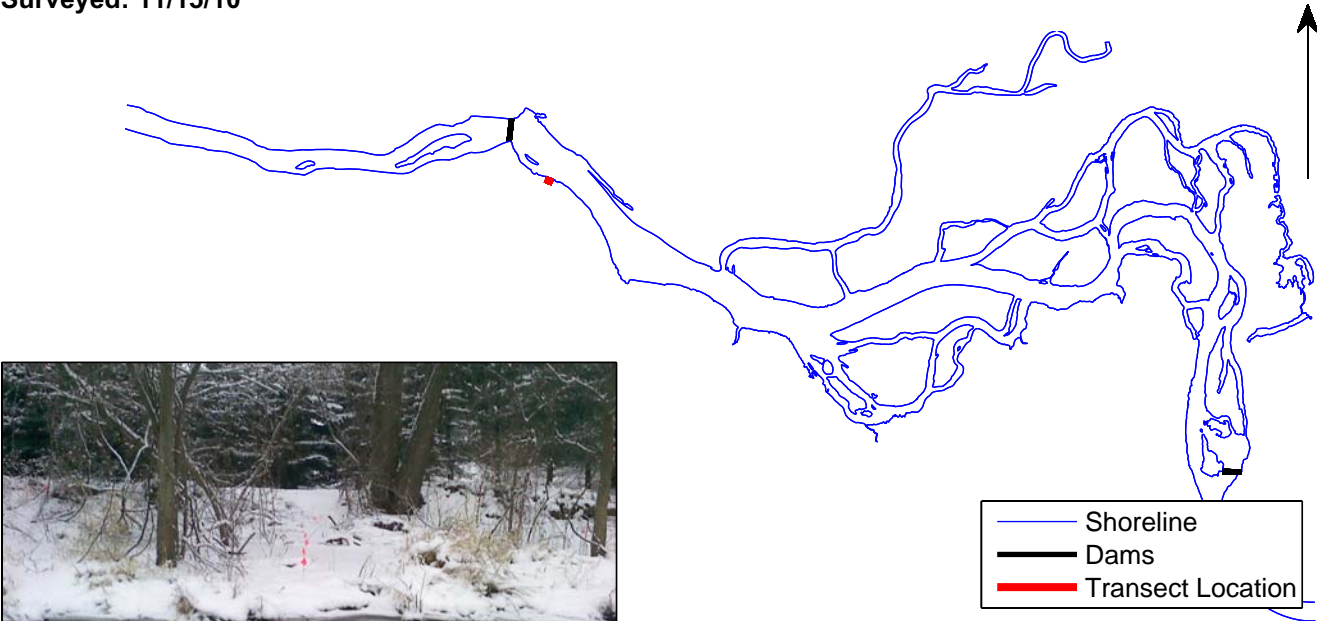
Otsego City Area 2 Bank Profiles
Transect: OCBN-32
Surveyed: 12/16/10

DRAFT



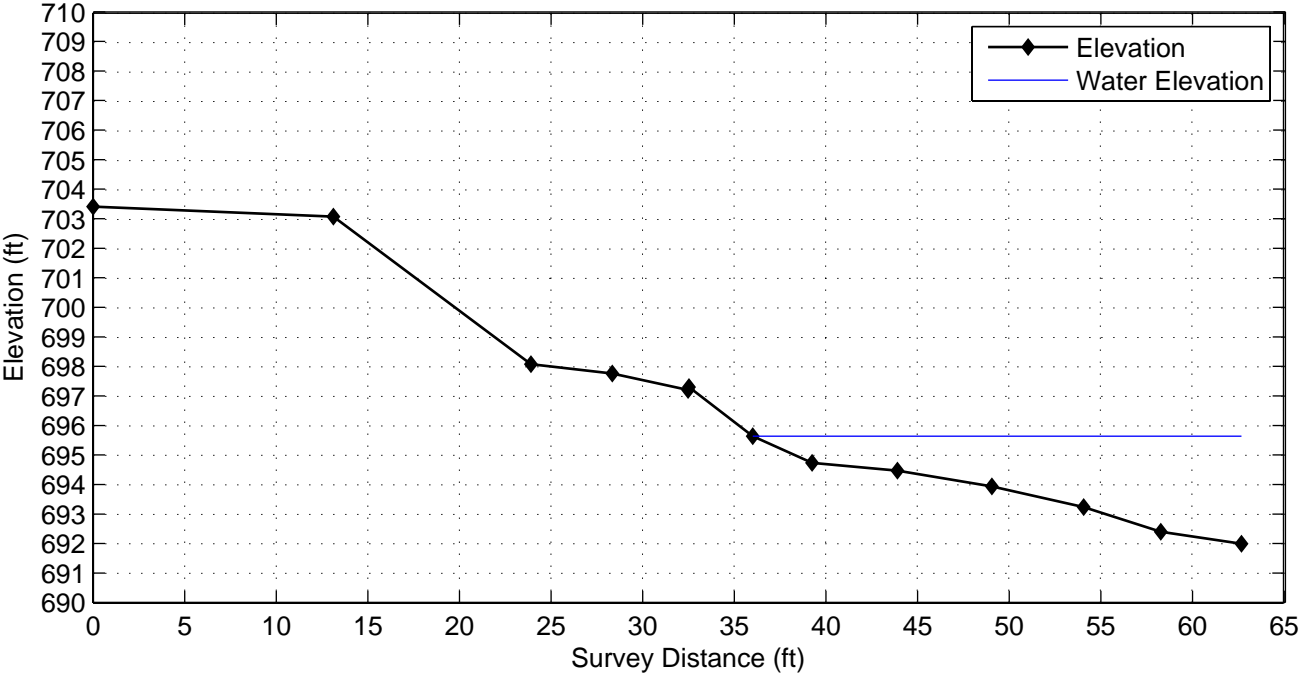
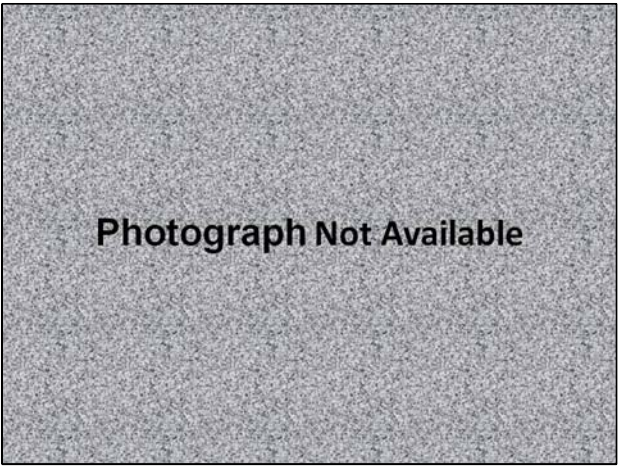
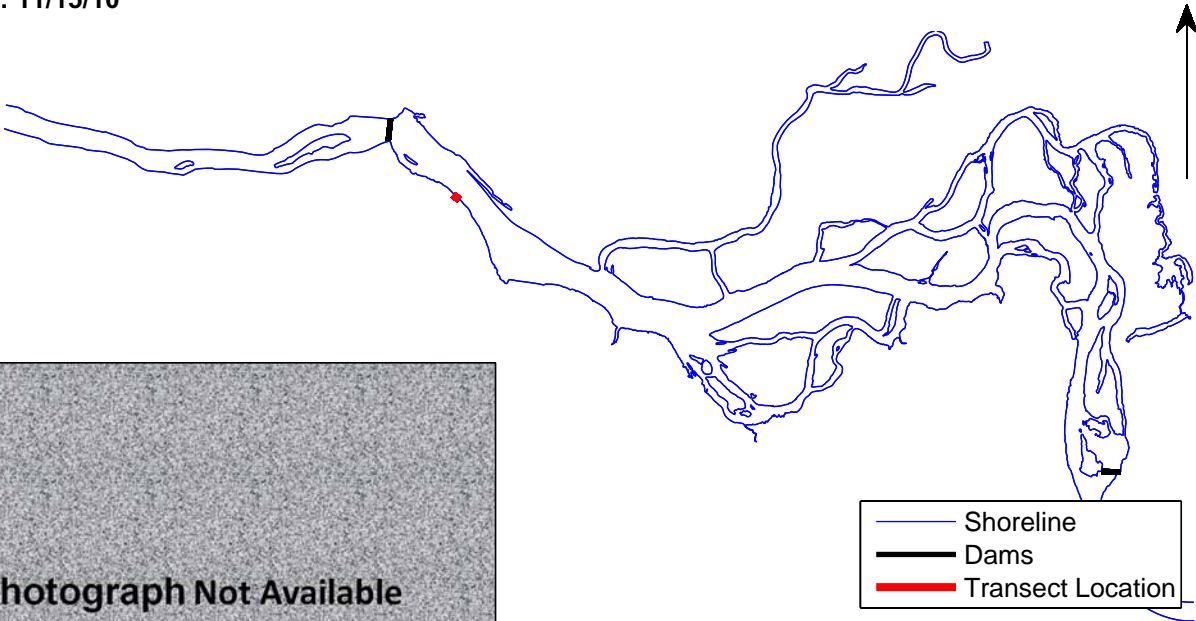
Otsego City Area 2 Bank Profiles
Transect: OCBS-01
Surveyed: 11/15/10

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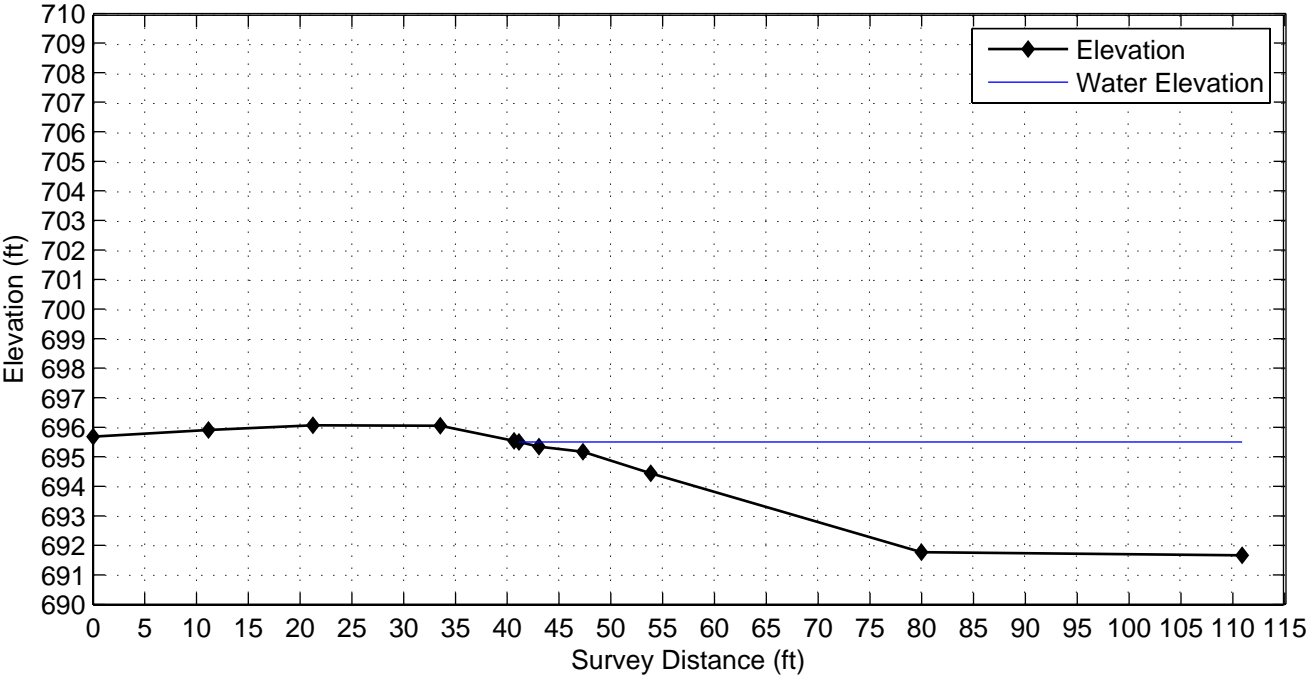
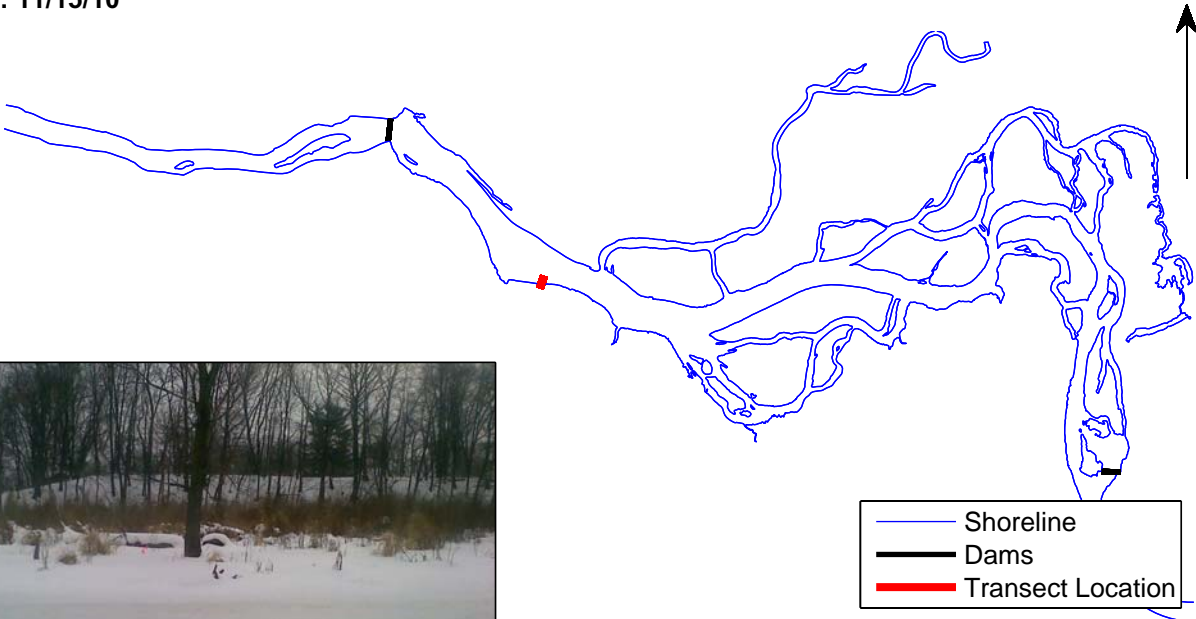
Otsego City Area 2 Bank Profiles
Transect: OCBS-02
Surveyed: 11/15/10

DRAFT



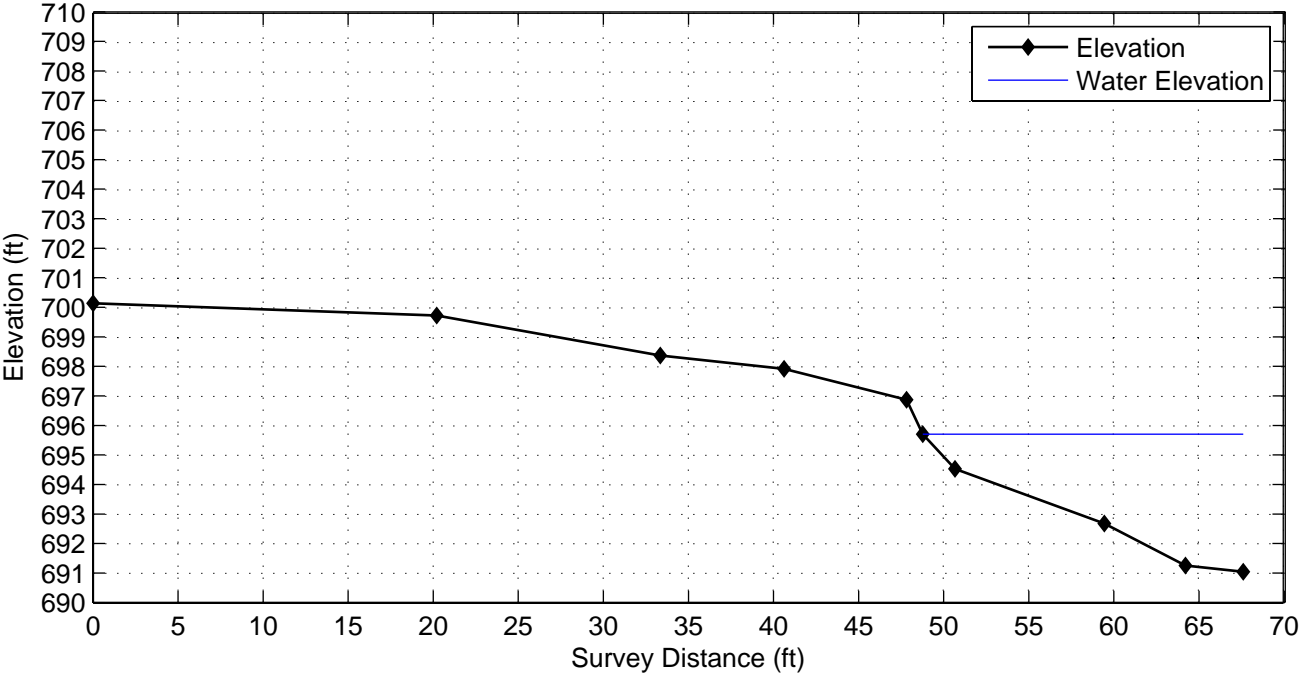
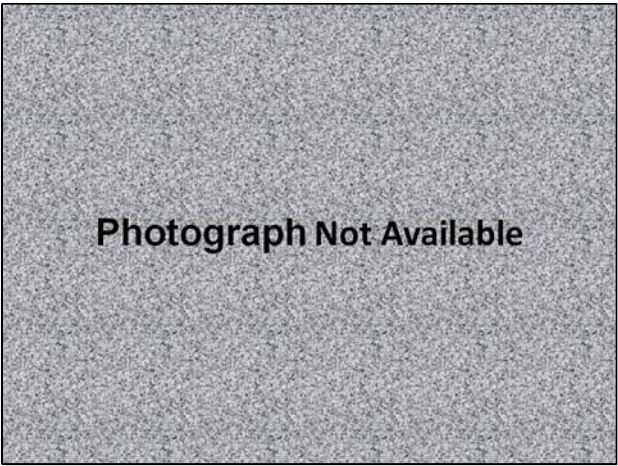
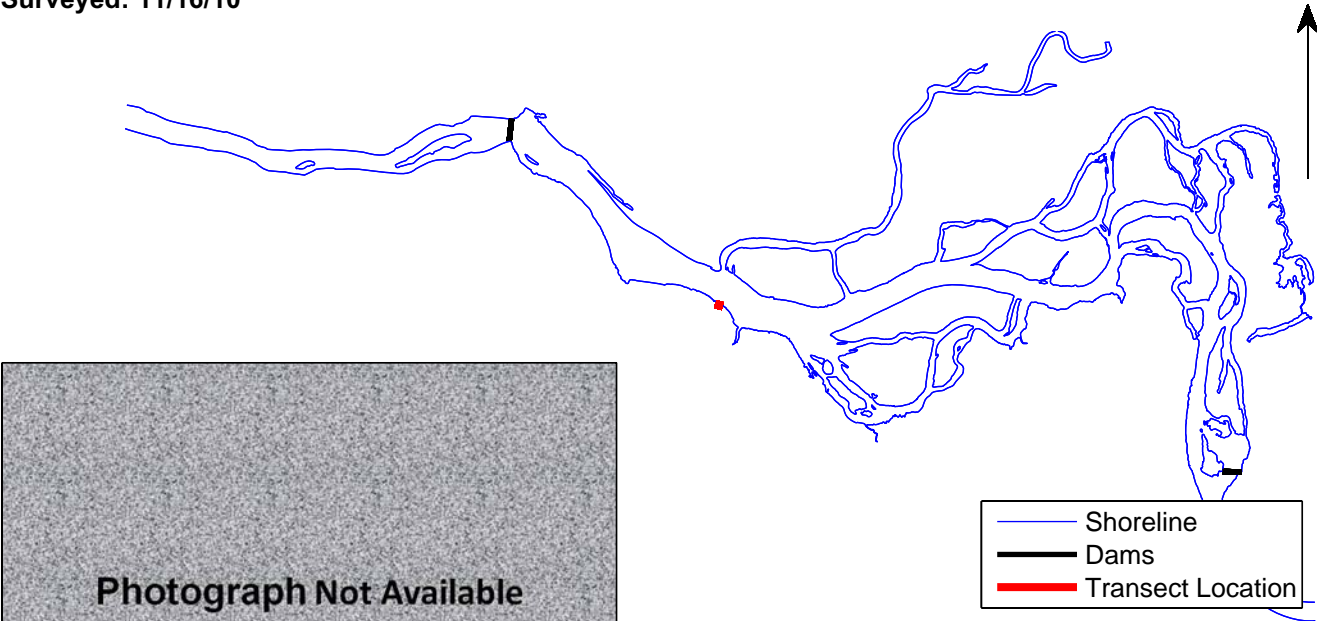
Otsego City Area 2 Bank Profiles
Transect: OCBS-04
Surveyed: 11/15/10

DRAFT



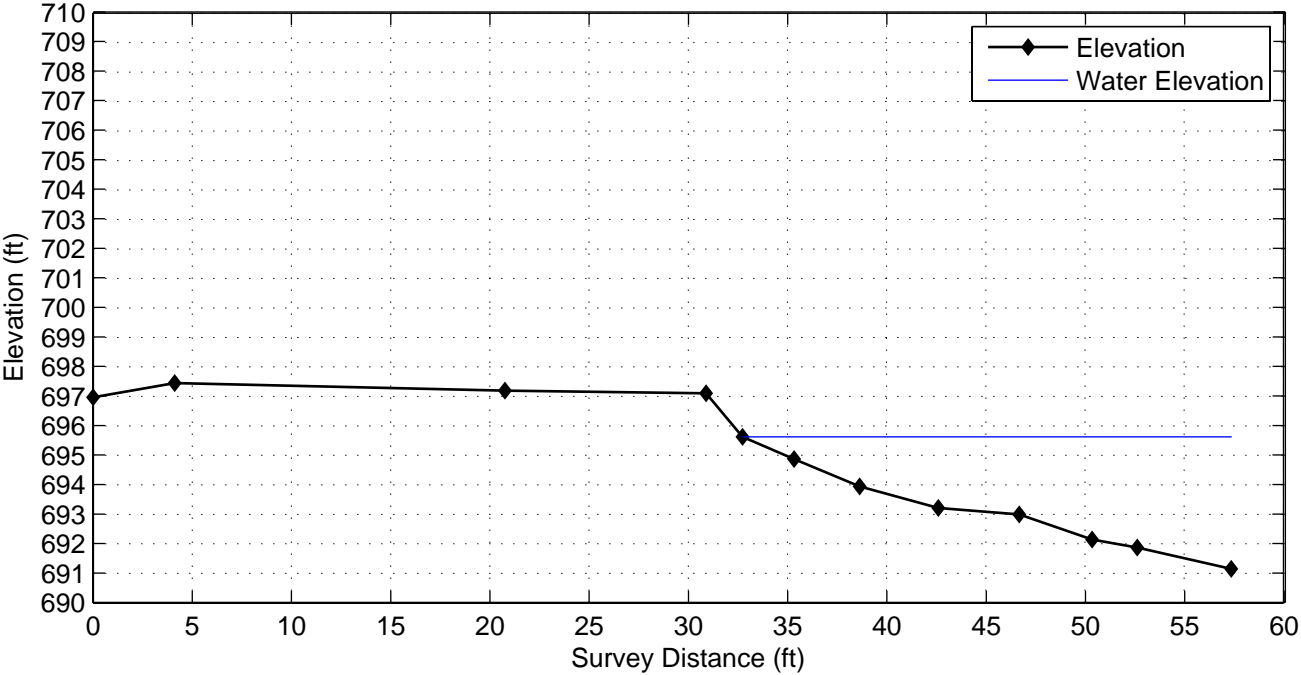
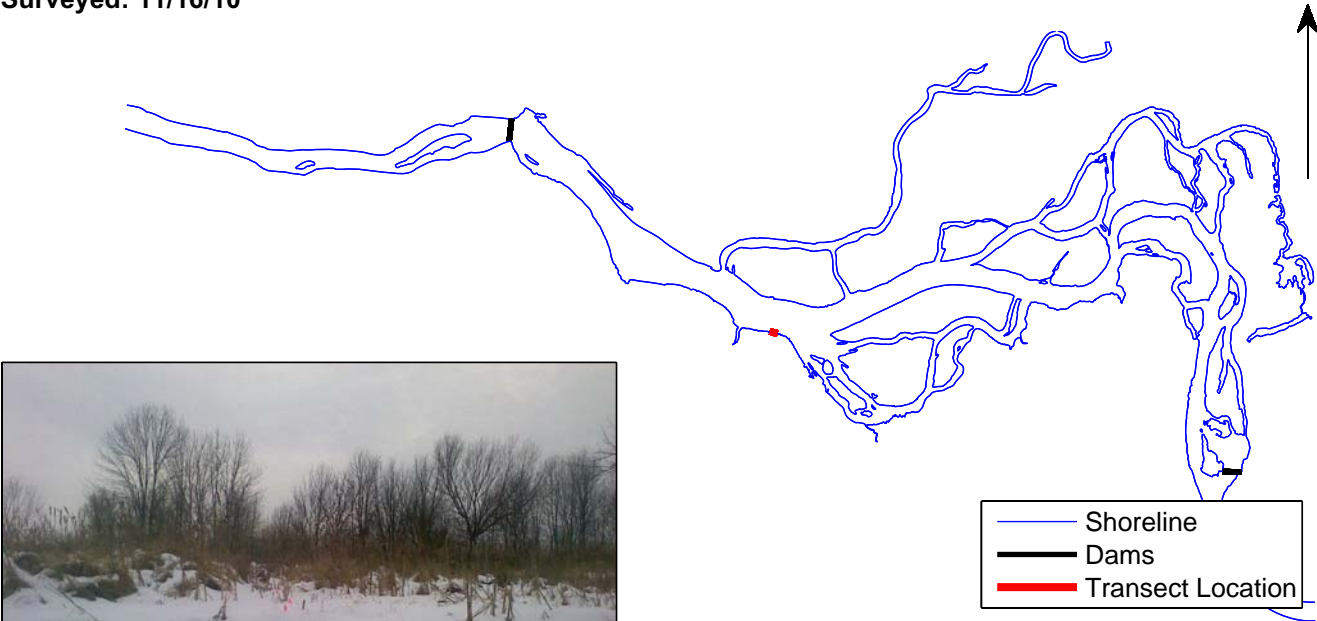
Otsego City Area 2 Bank Profiles
Transect: OCBS-05
Surveyed: 11/16/10

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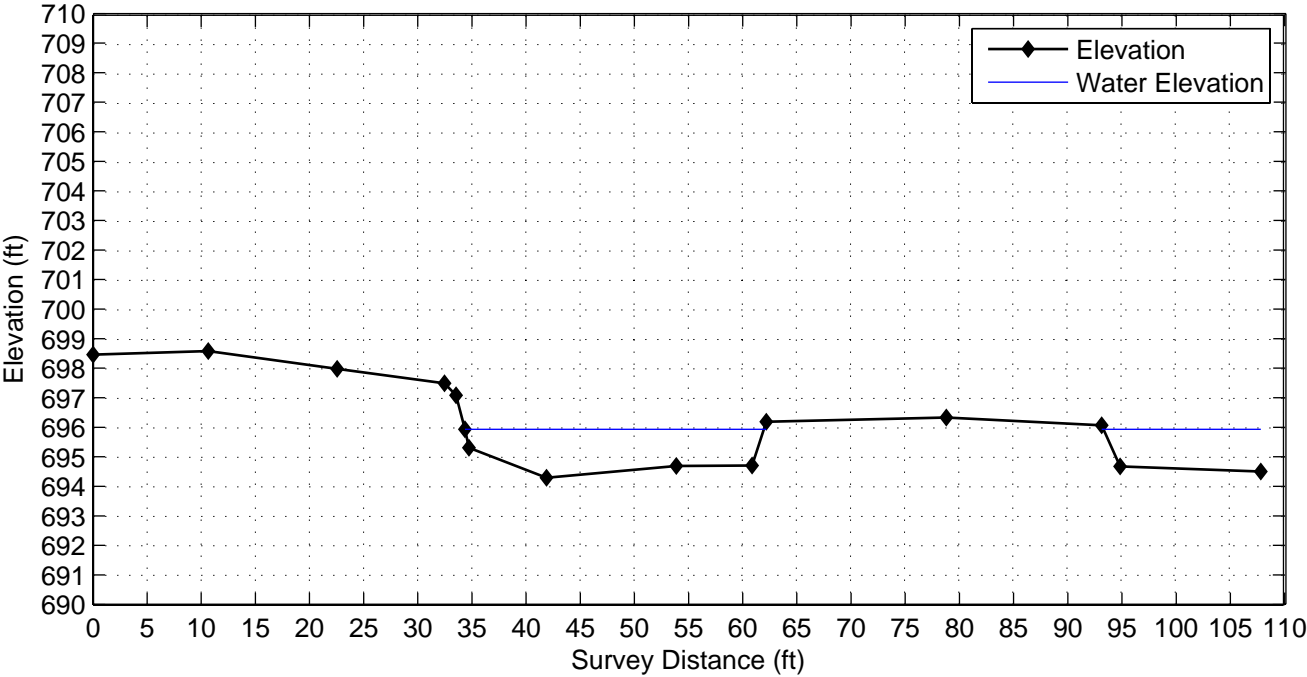
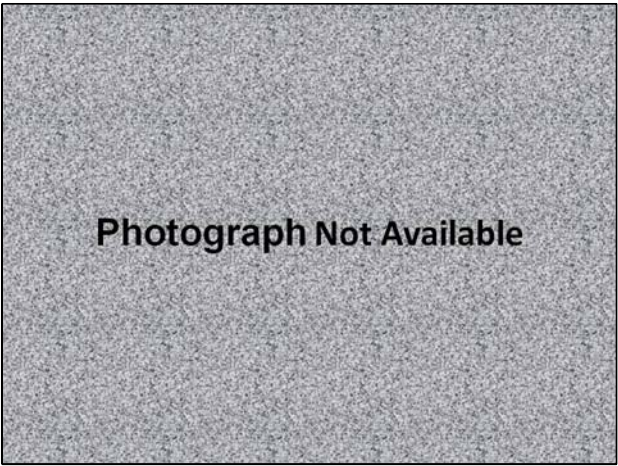
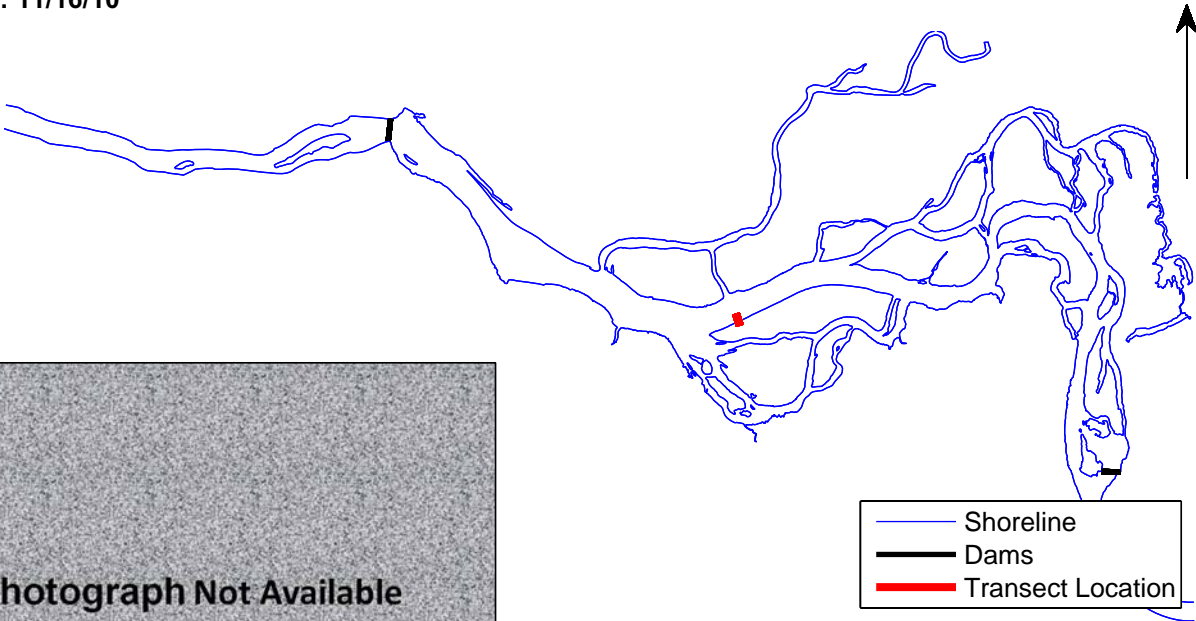
Otsego City Area 2 Bank Profiles
Transect: OCBS-06
Surveyed: 11/16/10

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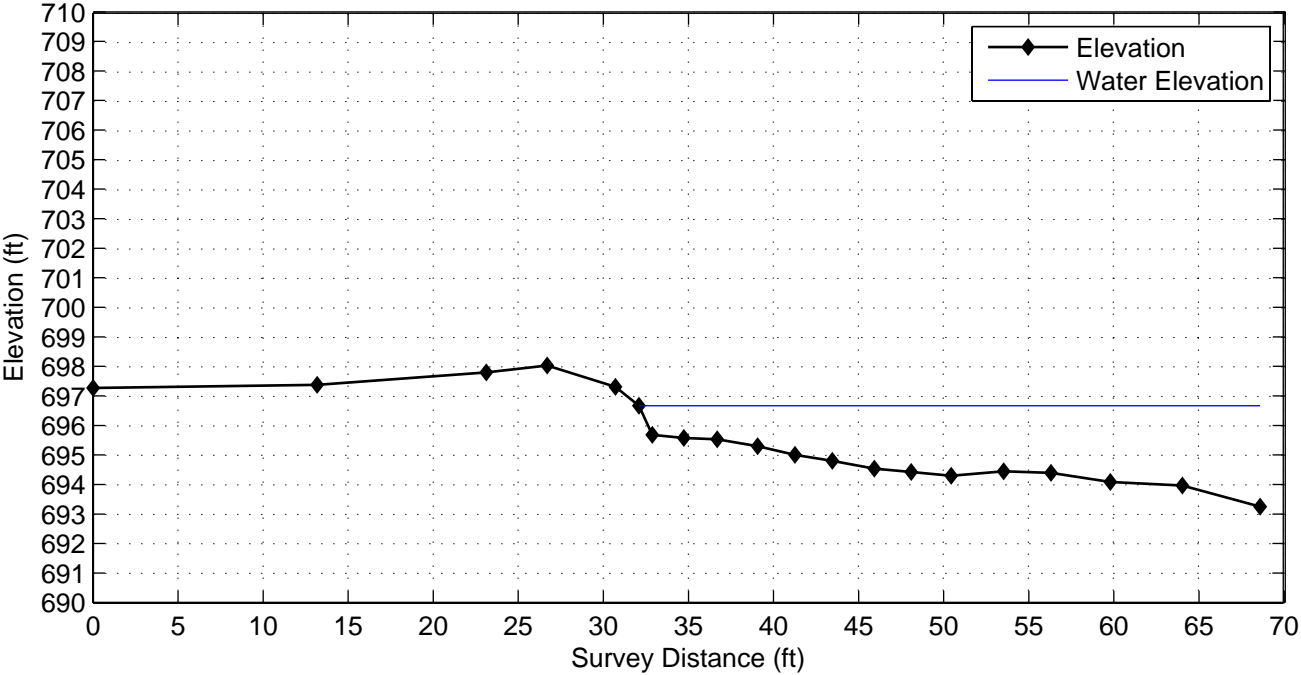
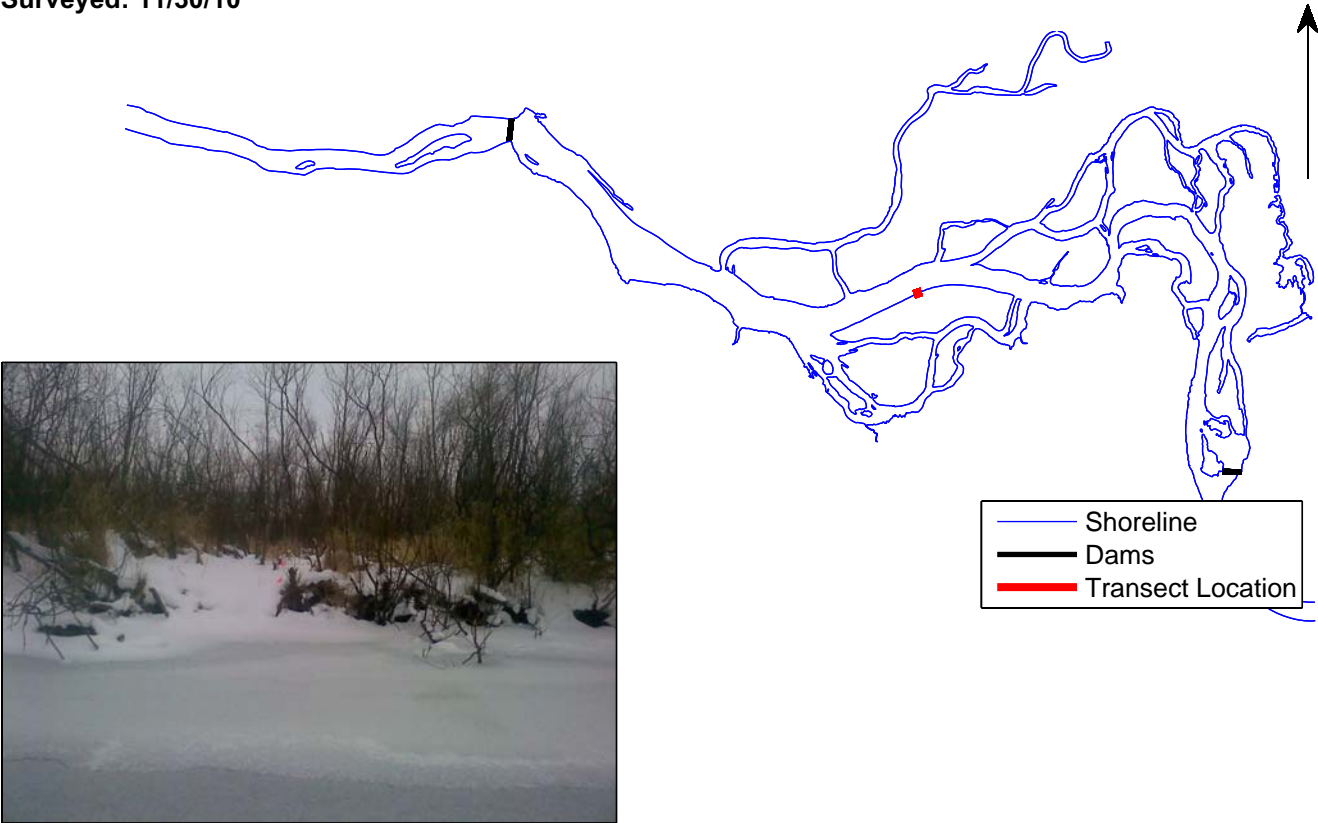
Otsego City Area 2 Bank Profiles
Transect: OCBS-07
Surveyed: 11/16/10

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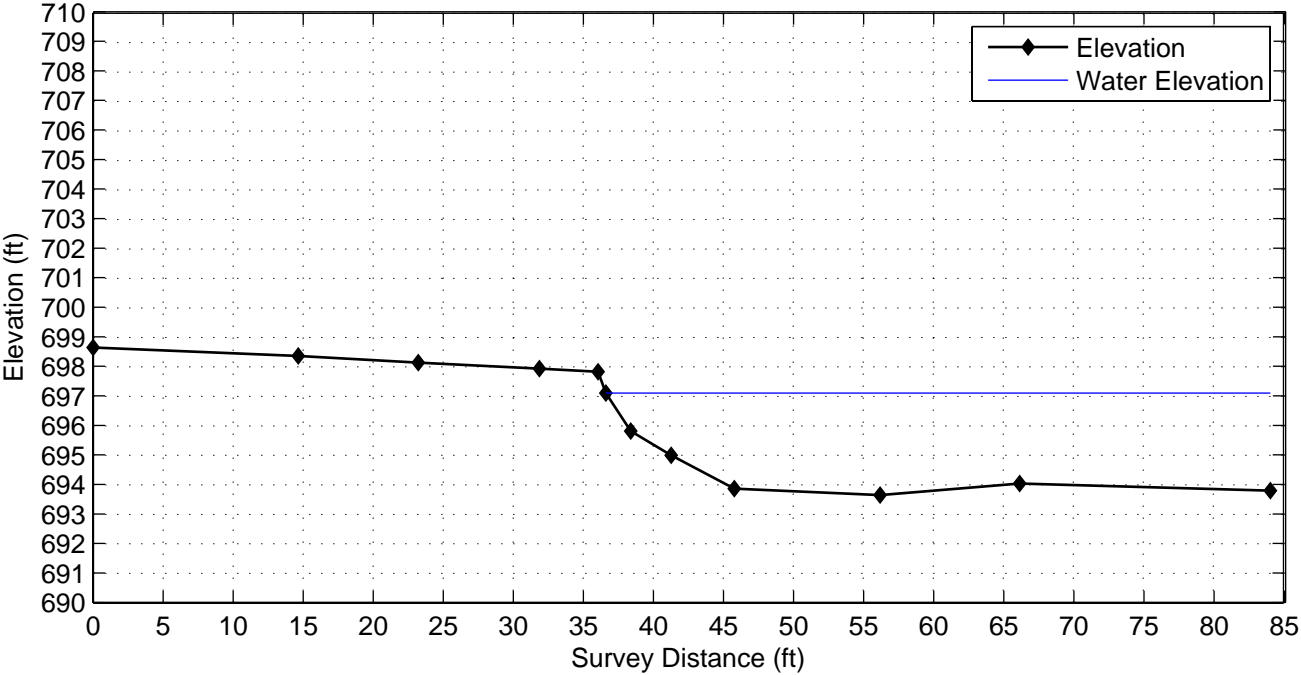
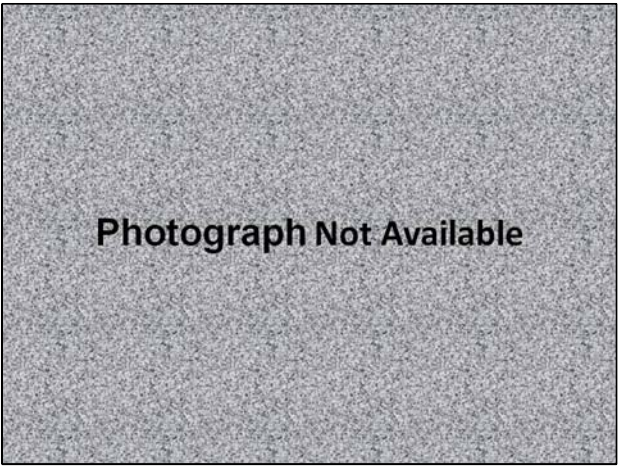
Otsego City Area 2 Bank Profiles
Transect: OCBS-08
Surveyed: 11/30/10

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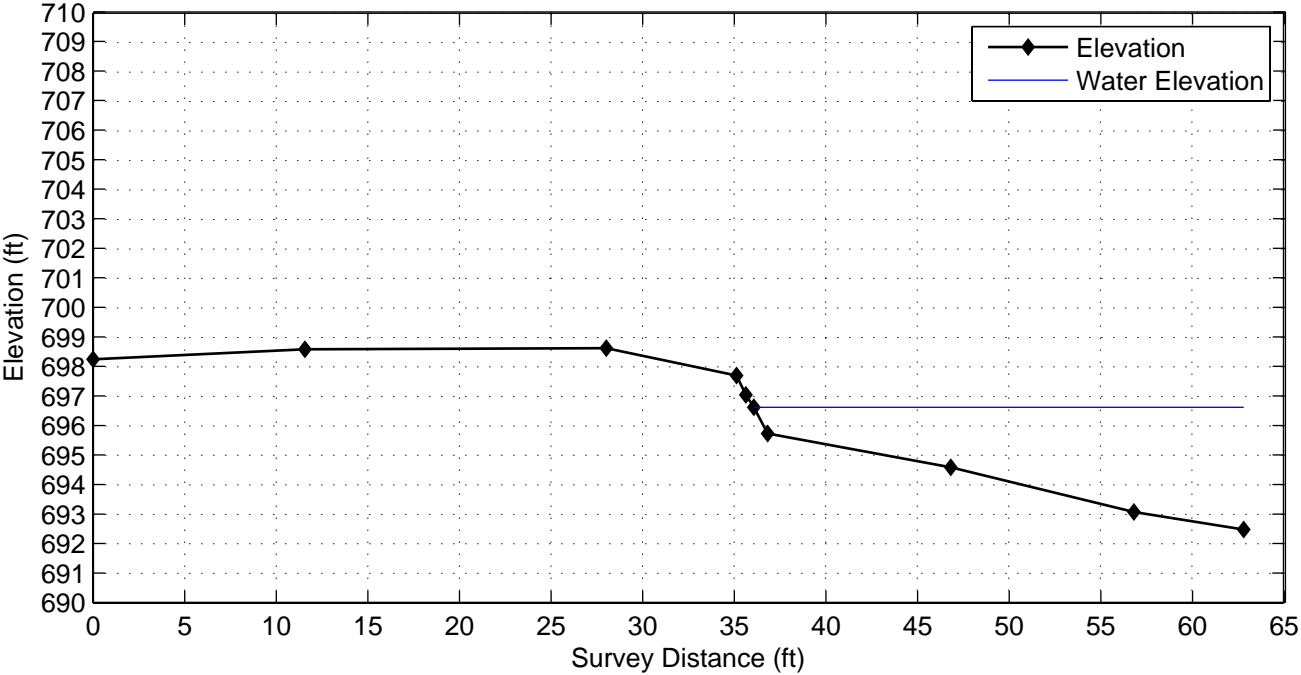
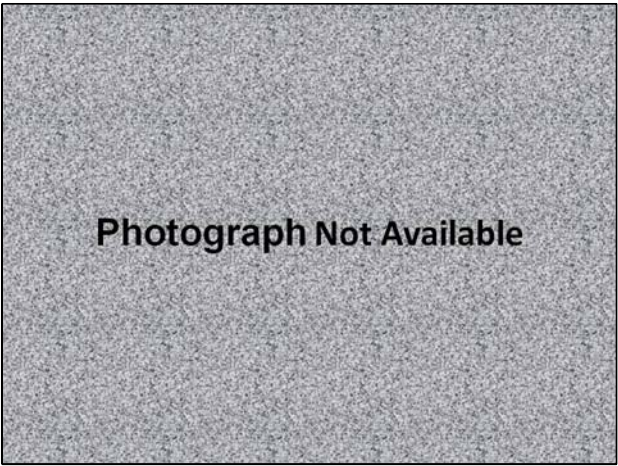
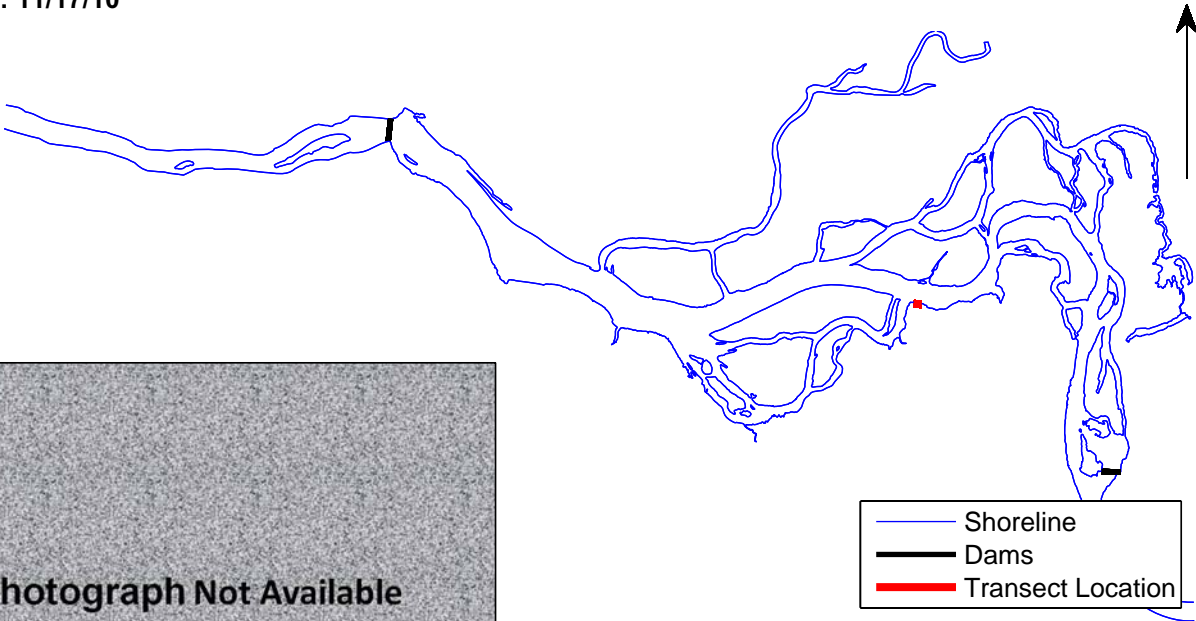
Otsego City Area 2 Bank Profiles
Transect: OCBS-09
Surveyed: 11/30/10

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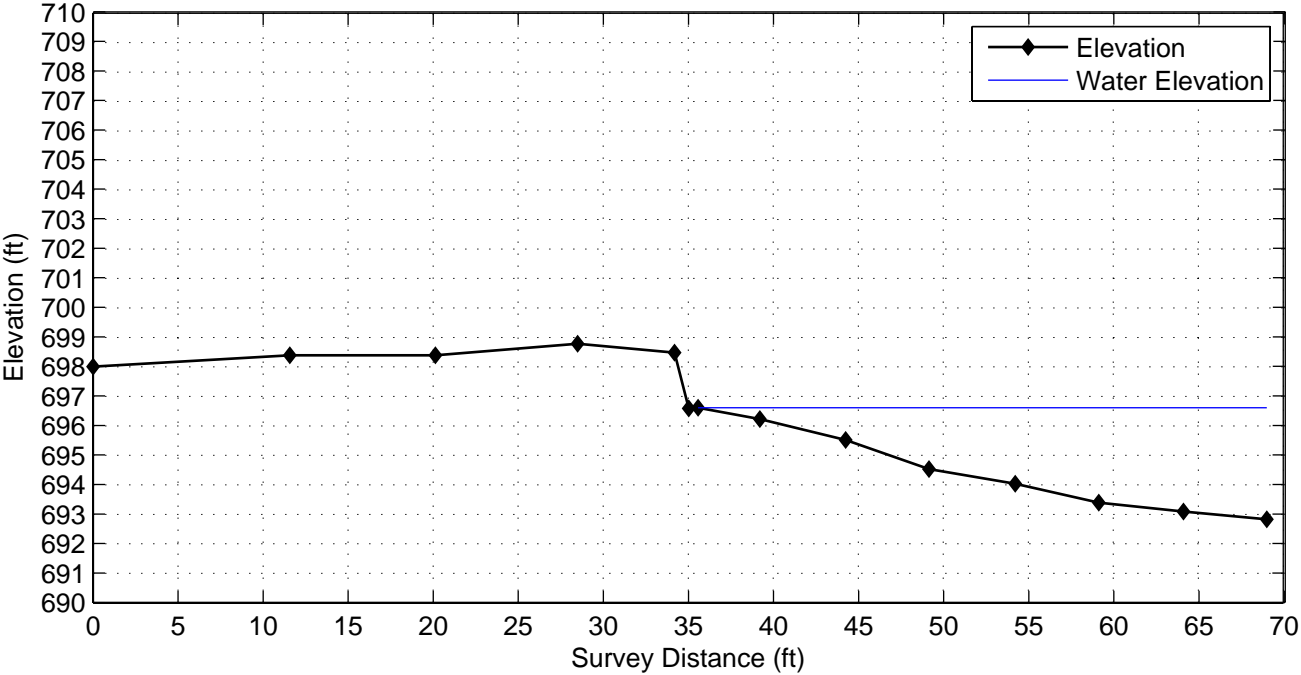
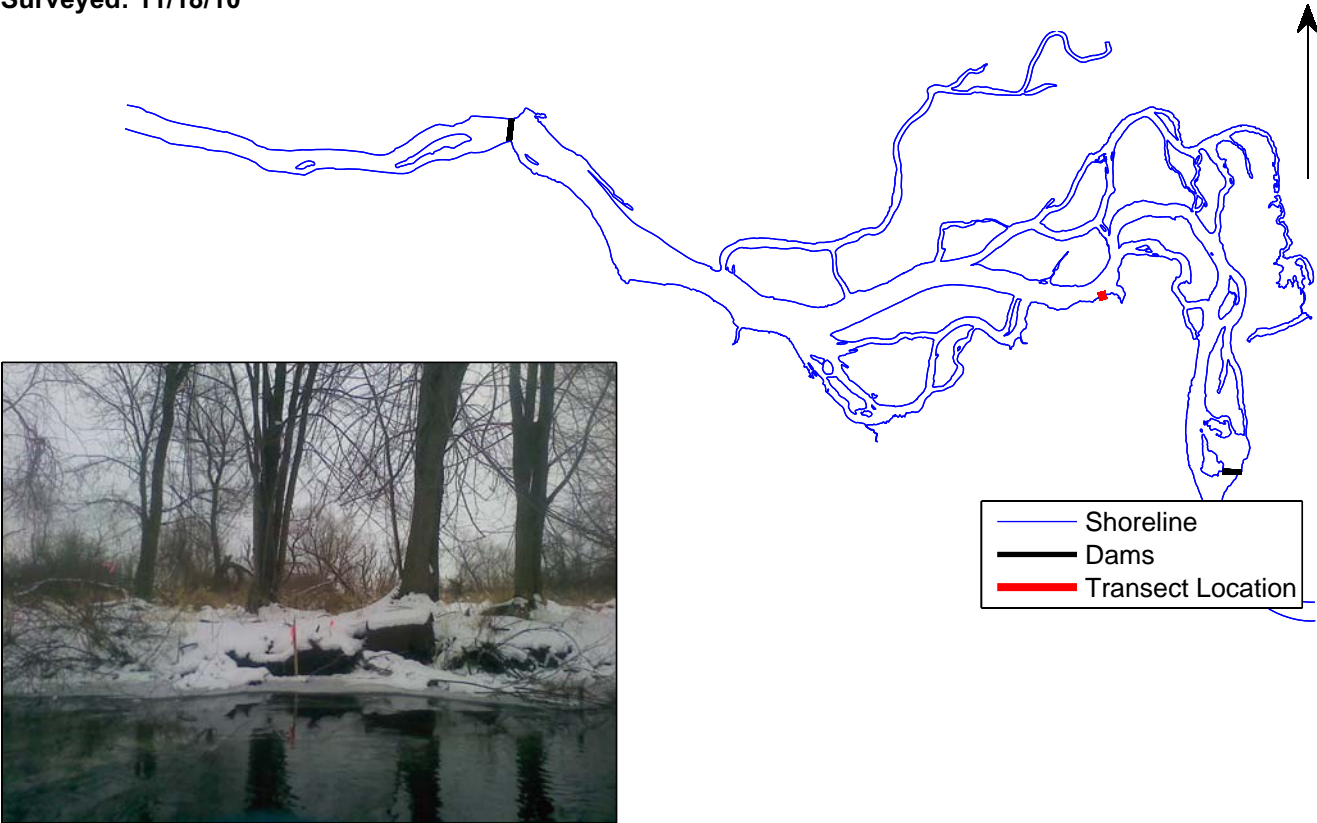
Otsego City Area 2 Bank Profiles
Transect: OCBS-10
Surveyed: 11/17/10

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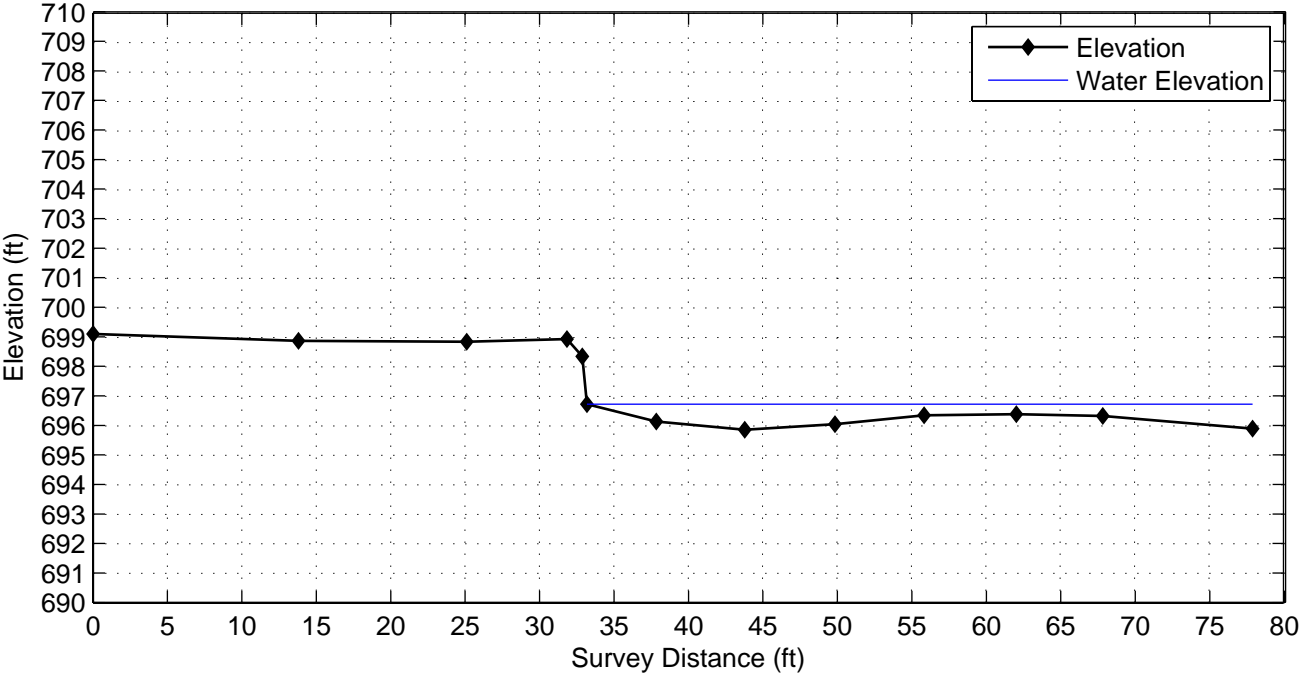
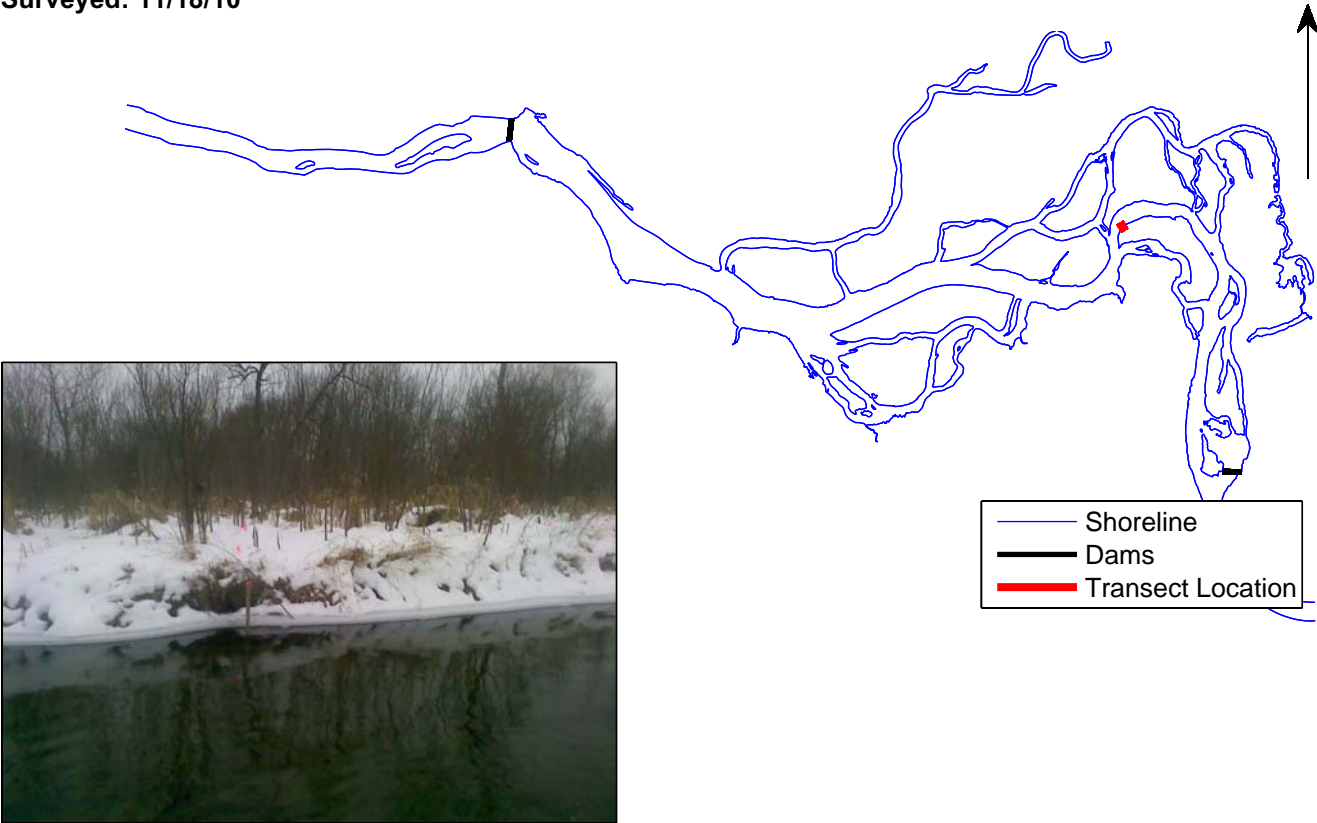
Otsego City Area 2 Bank Profiles
Transect: OCBS-11
Surveyed: 11/18/10

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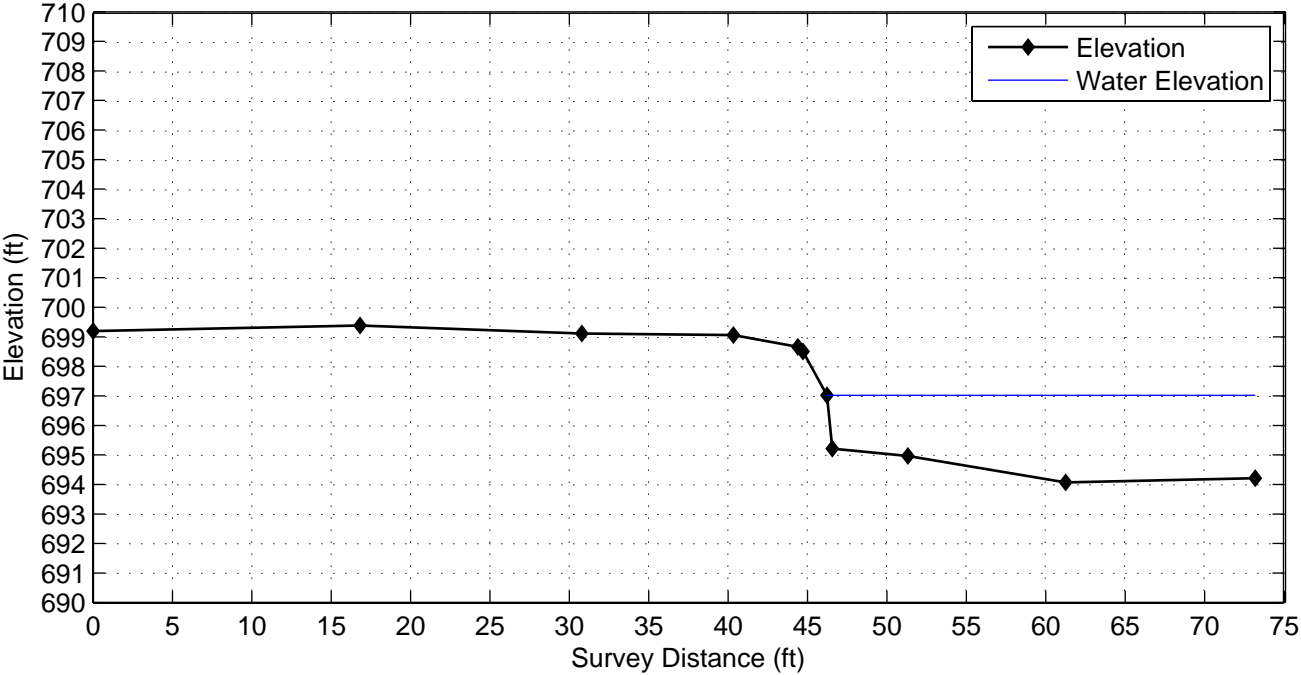
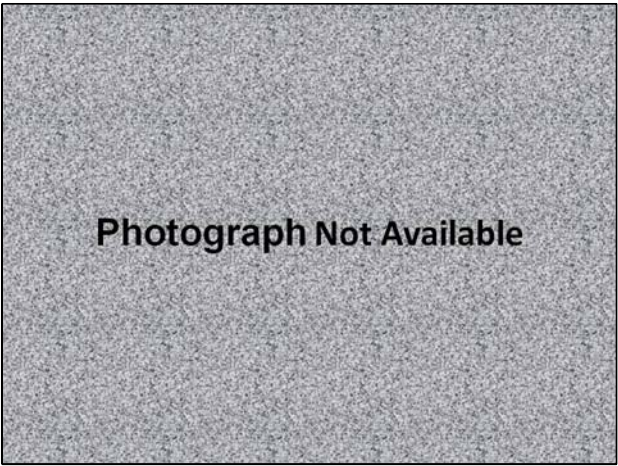
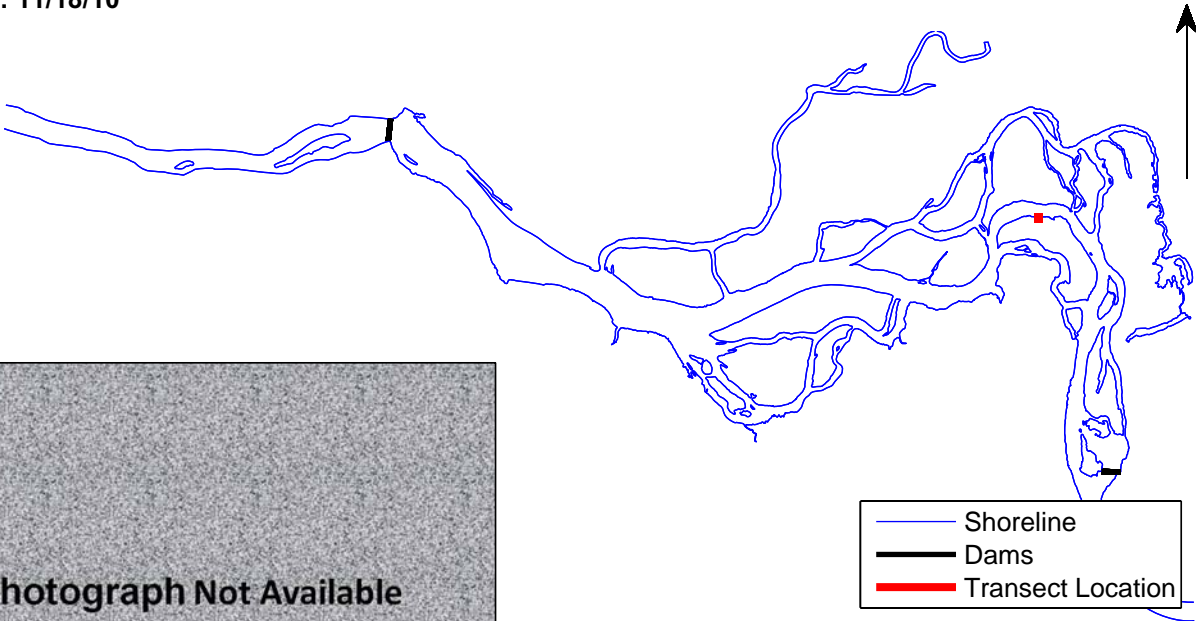
Otsego City Area 2 Bank Profiles
Transect: OCBS-12
Surveyed: 11/18/10

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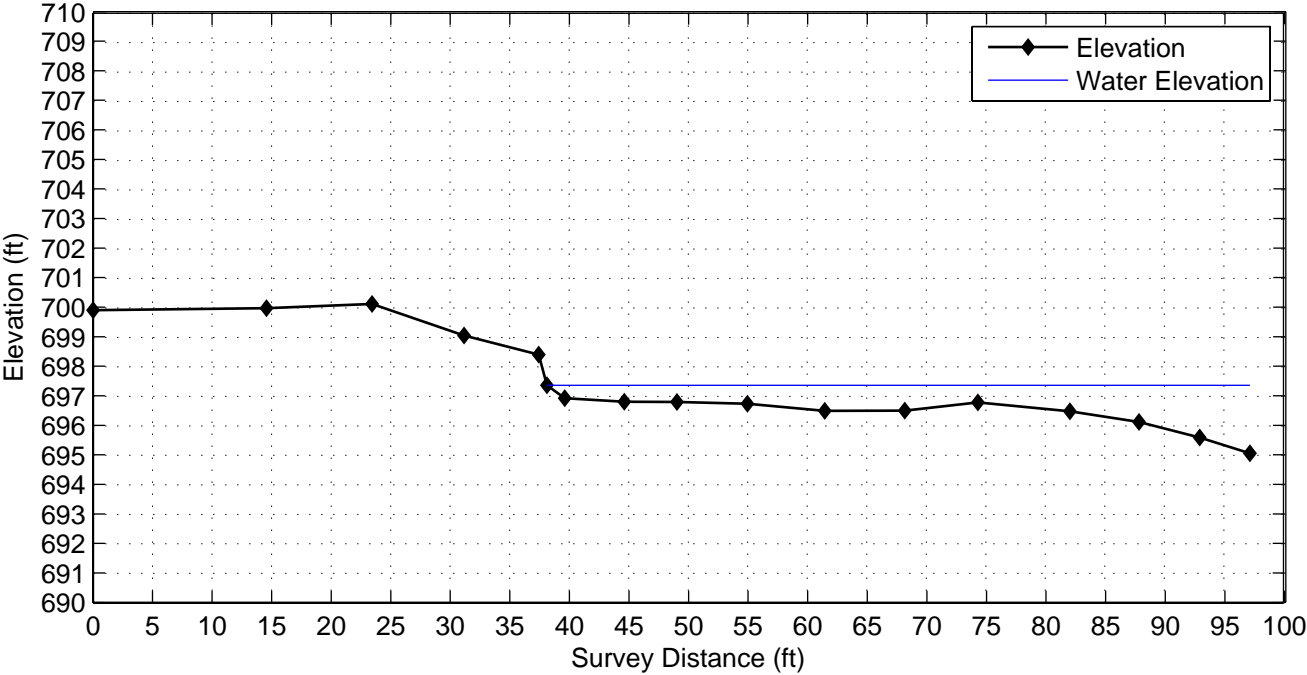
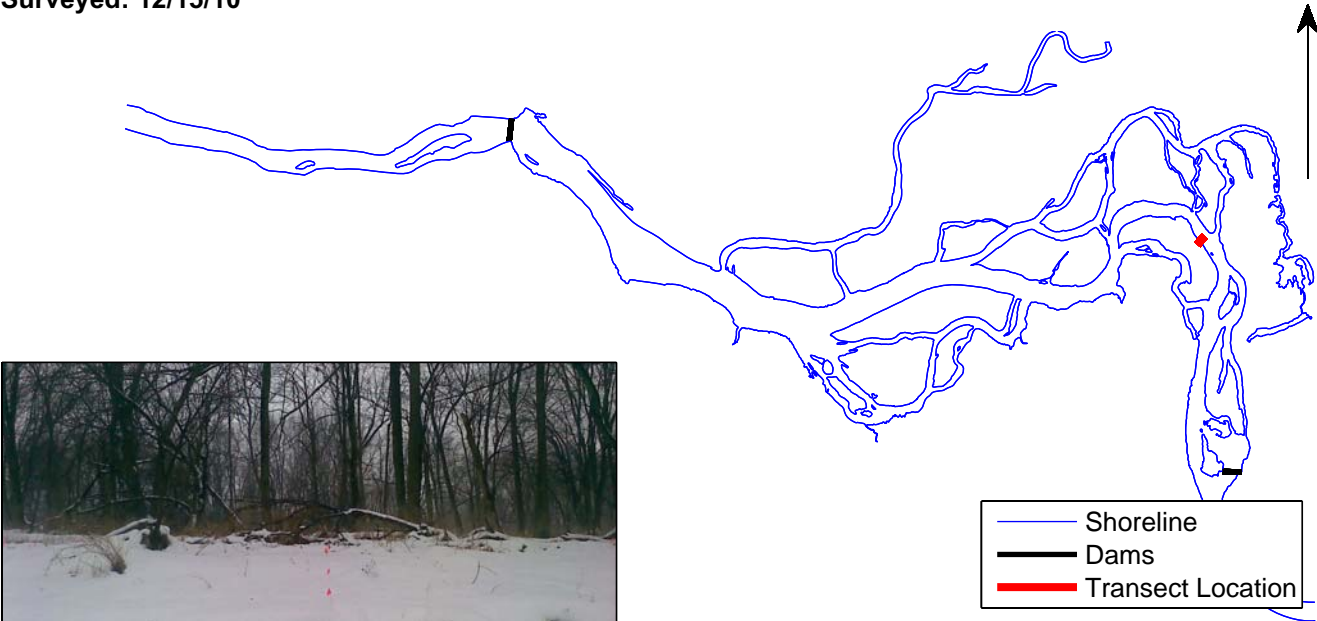
Otsego City Area 2 Bank Profiles
Transect: OCBS-13
Surveyed: 11/18/10

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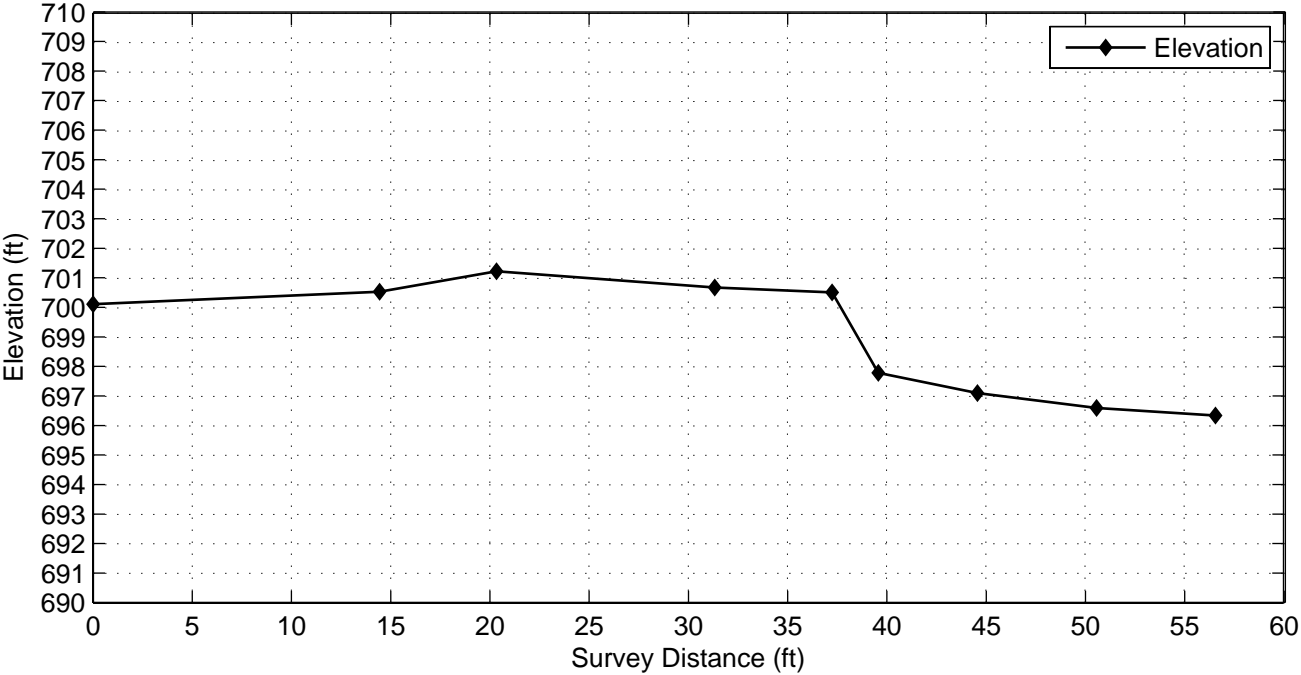
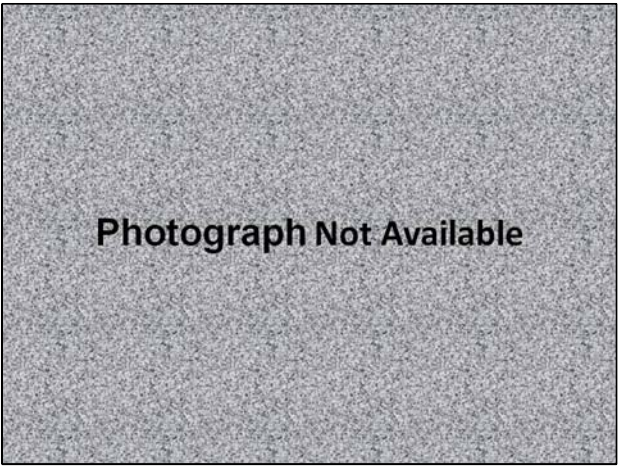
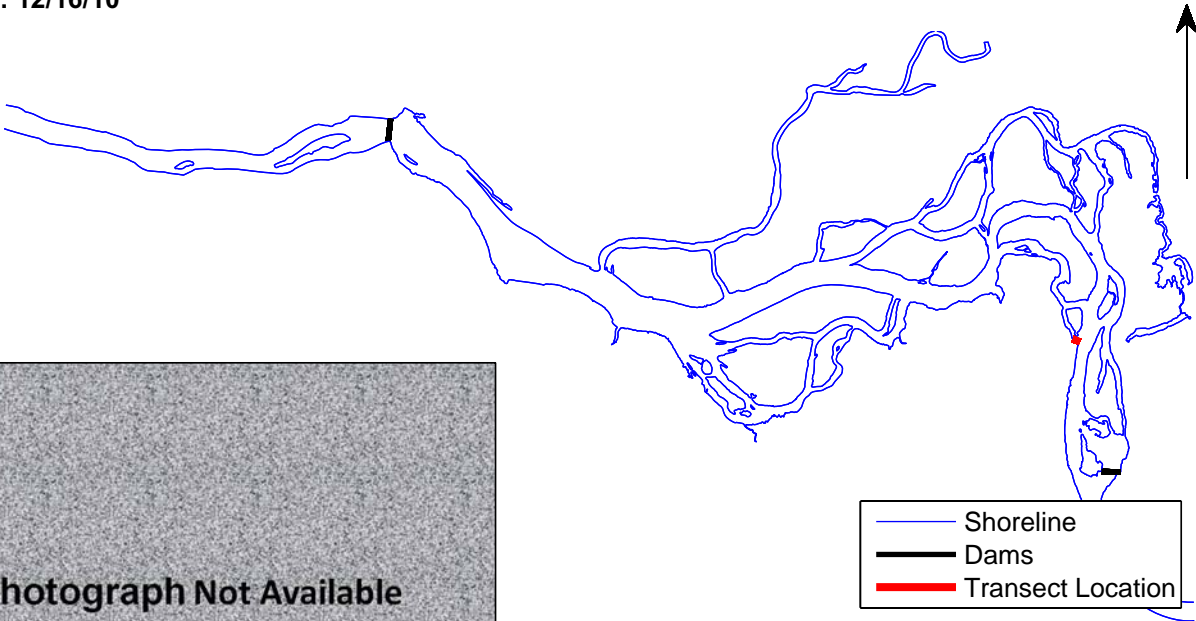
Otsego City Area 2 Bank Profiles
Transect: OCBS-14
Surveyed: 12/15/10

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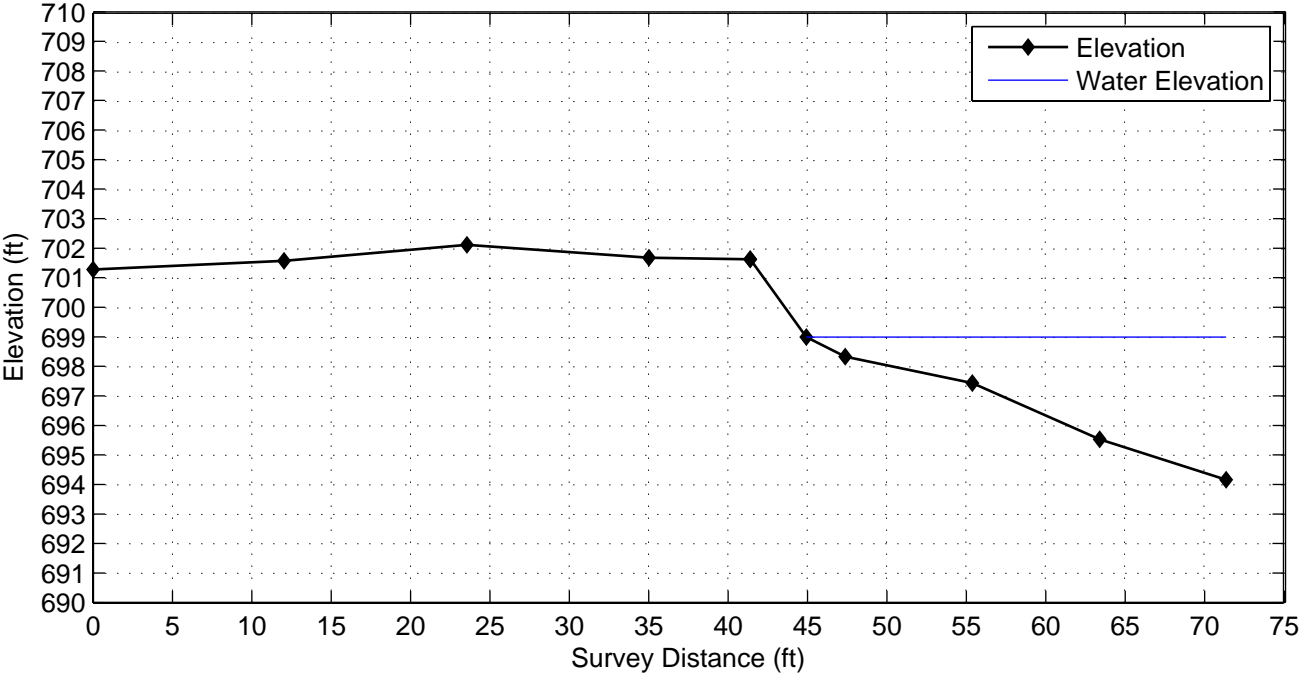
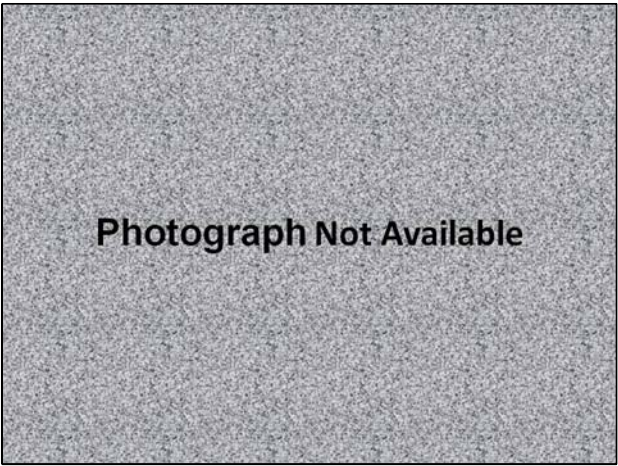
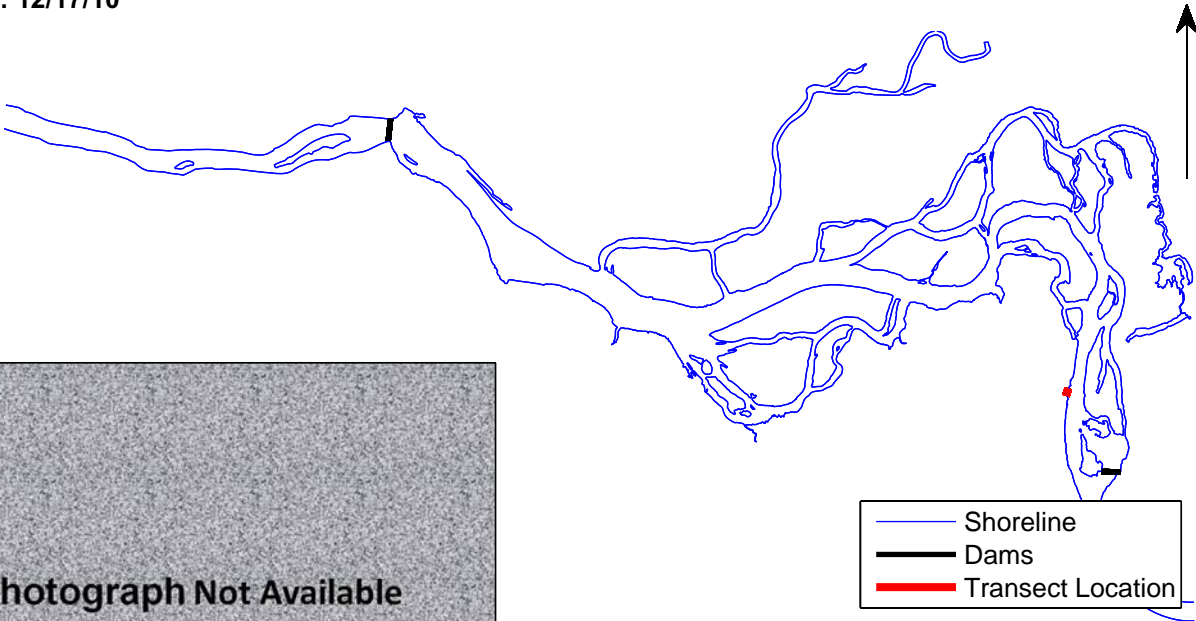
Otsego City Area 2 Bank Profiles
Transect: OCBS-15
Surveyed: 12/16/10

DRAFT



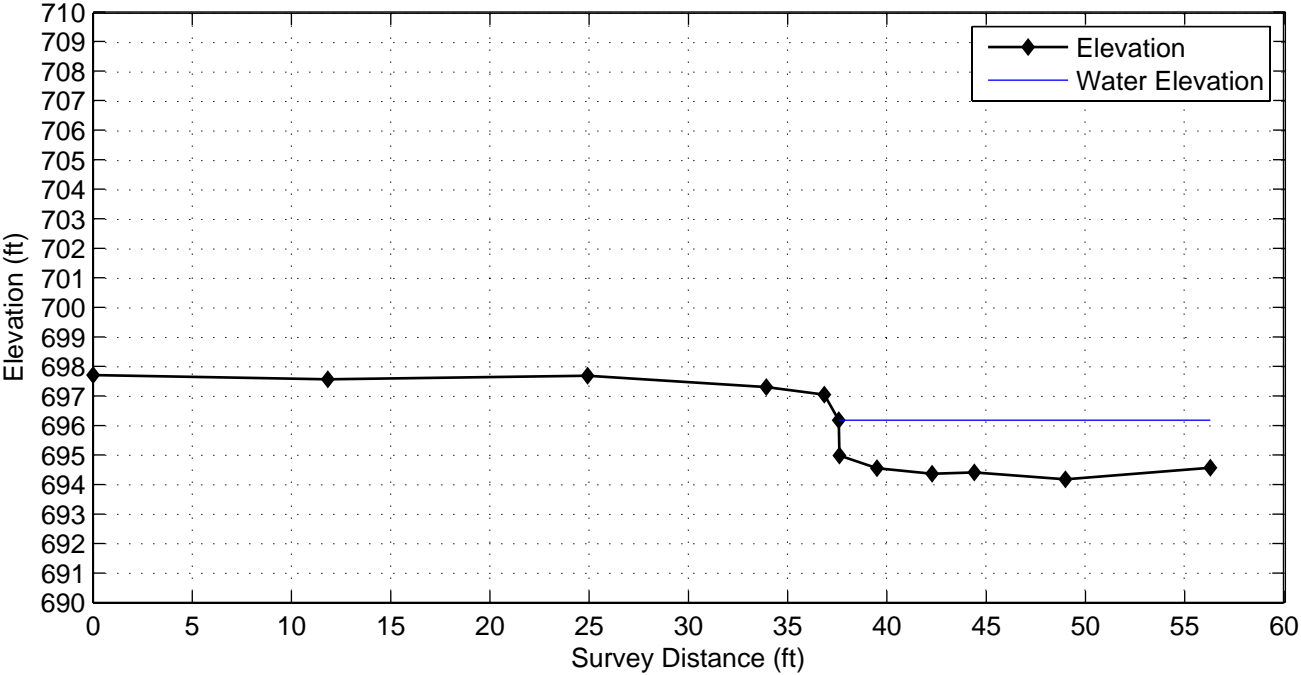
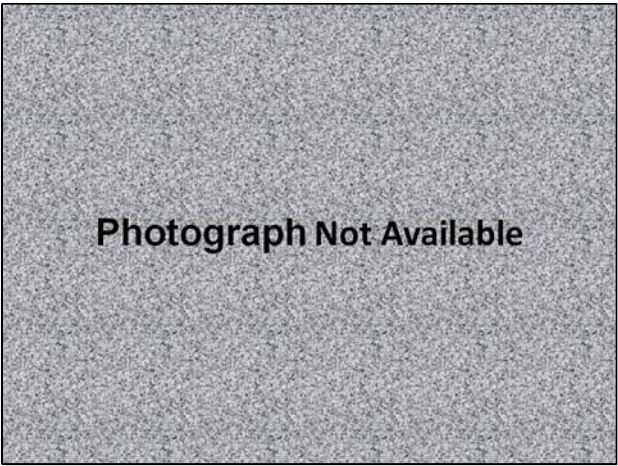
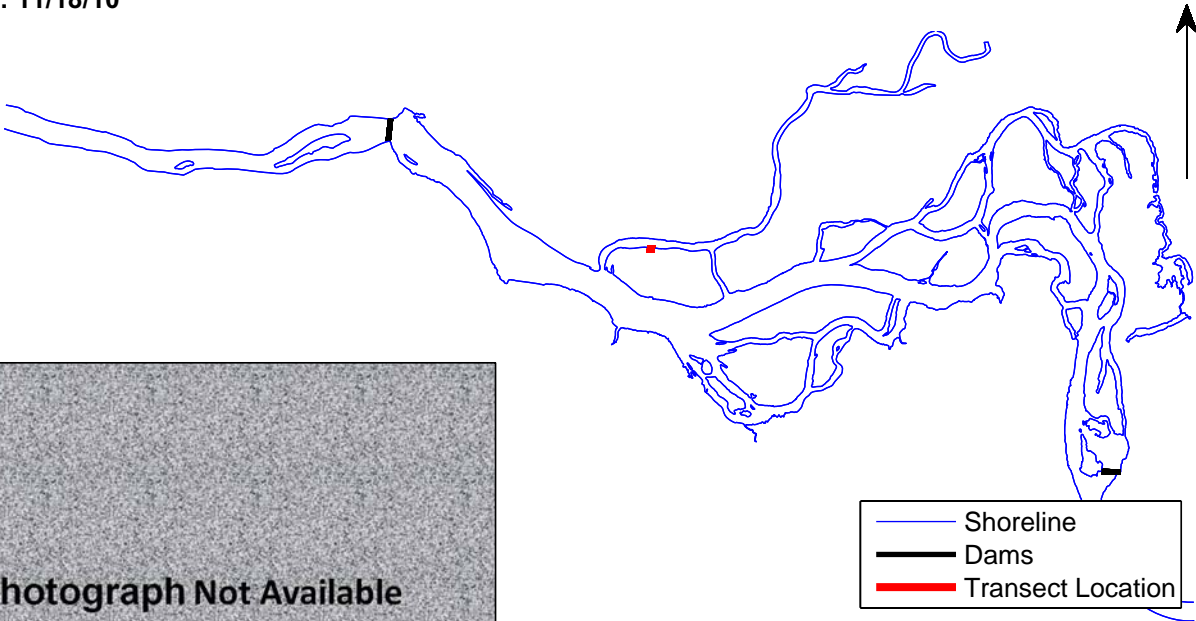
Otsego City Area 2 Bank Profiles
Transect: OCBS-16
Surveyed: 12/17/10

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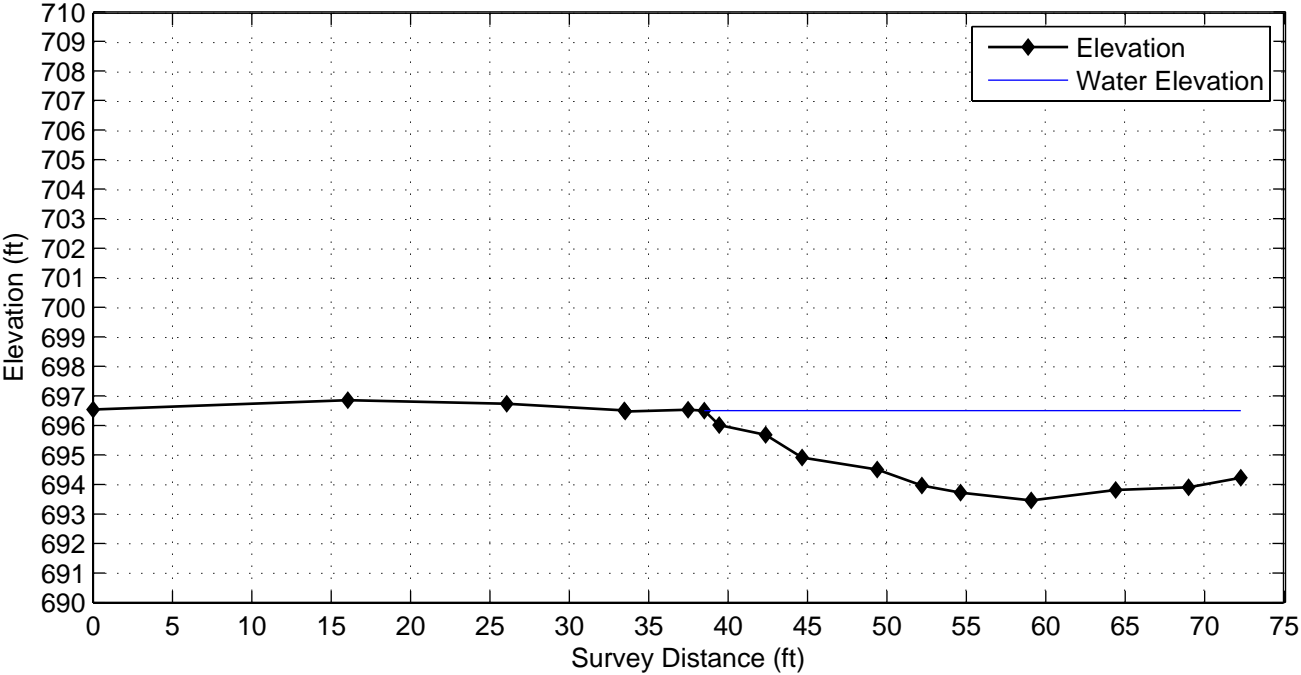
Otsego City Area 2 Bank Profiles
Transect: OCBS-17
Surveyed: 11/18/10

DRAFT



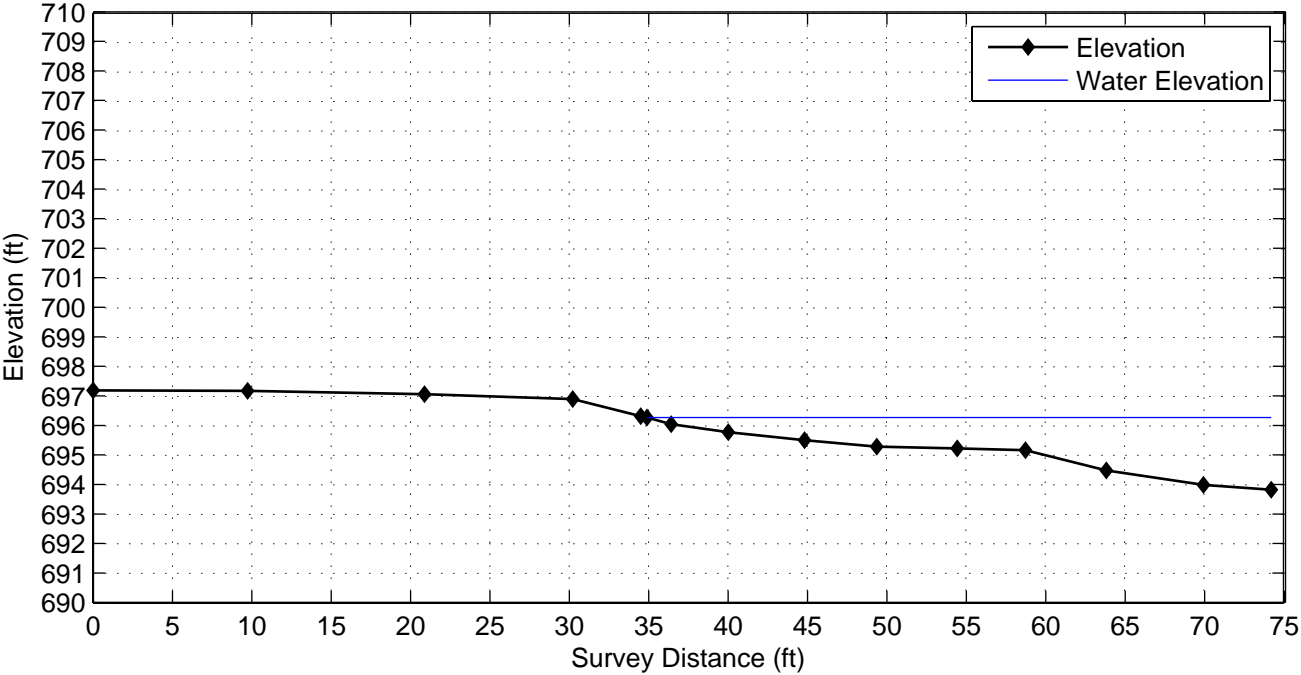
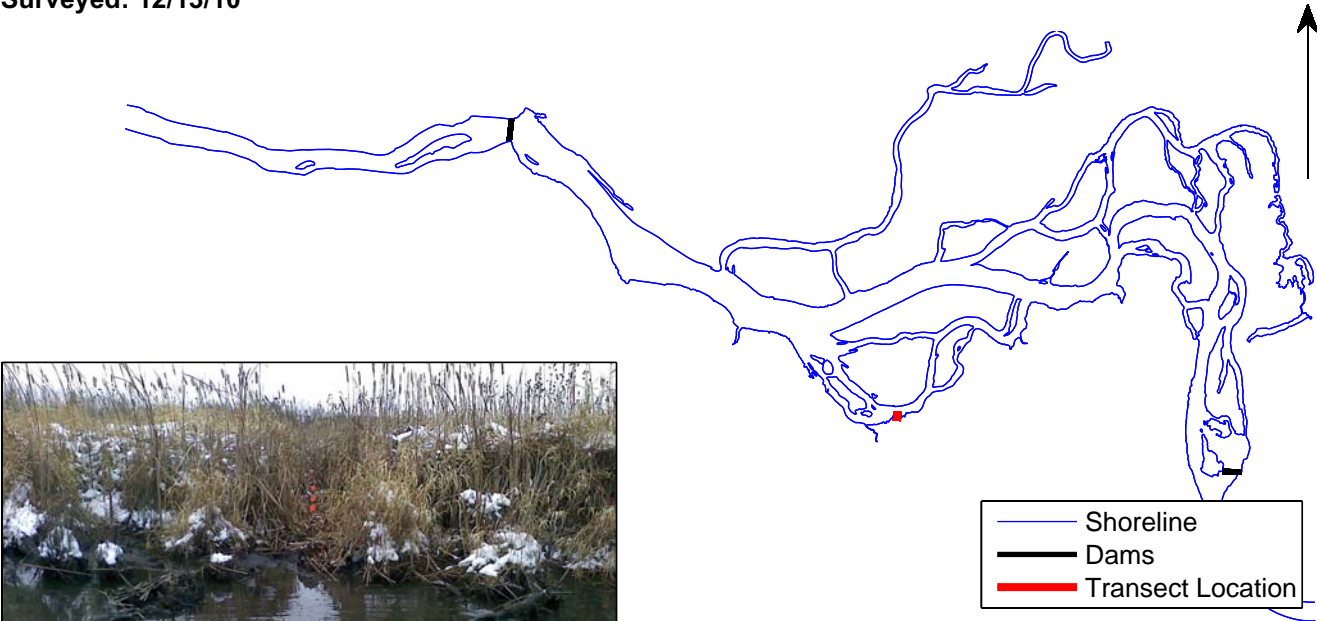
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Transect: OCBS-18
Surveyed: 12/01/10

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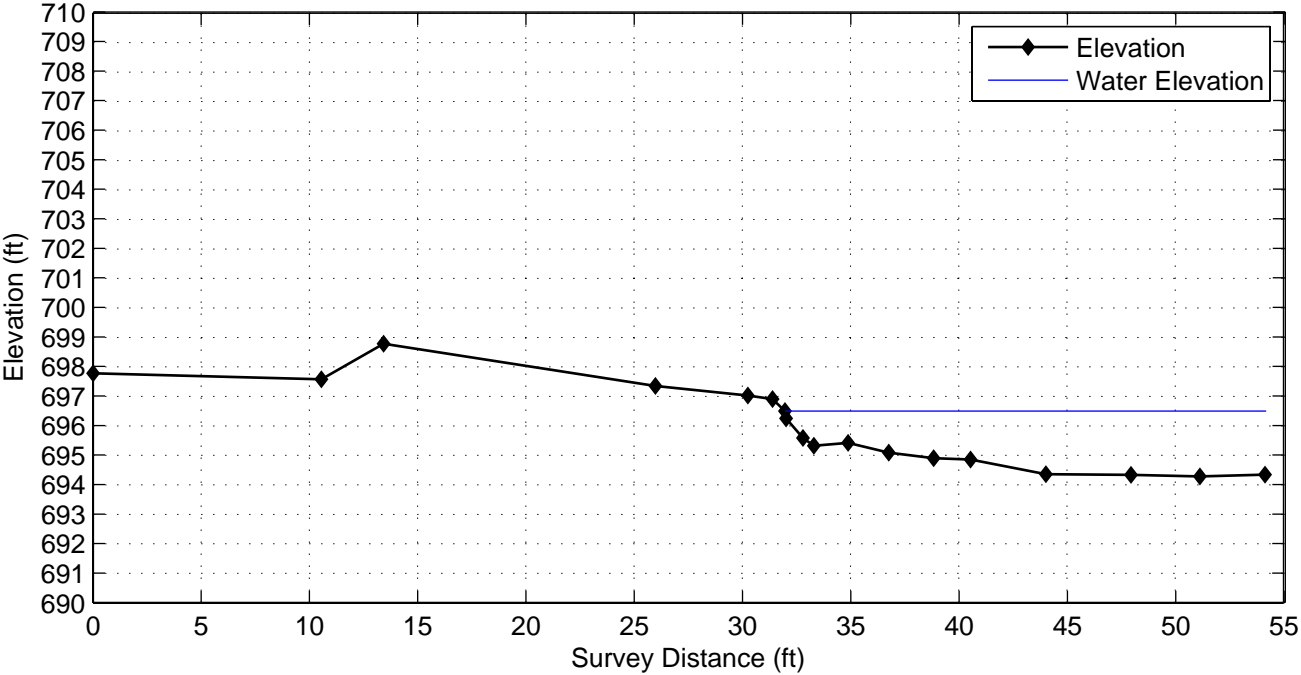
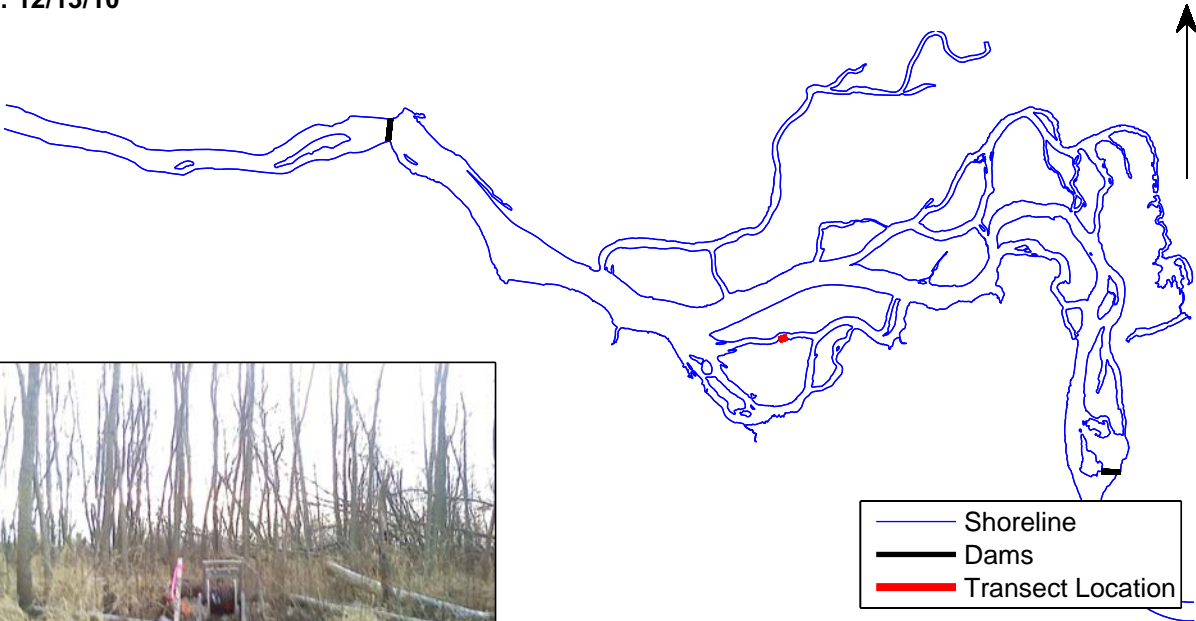
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Transect: OCBS-19
Surveyed: 12/13/10

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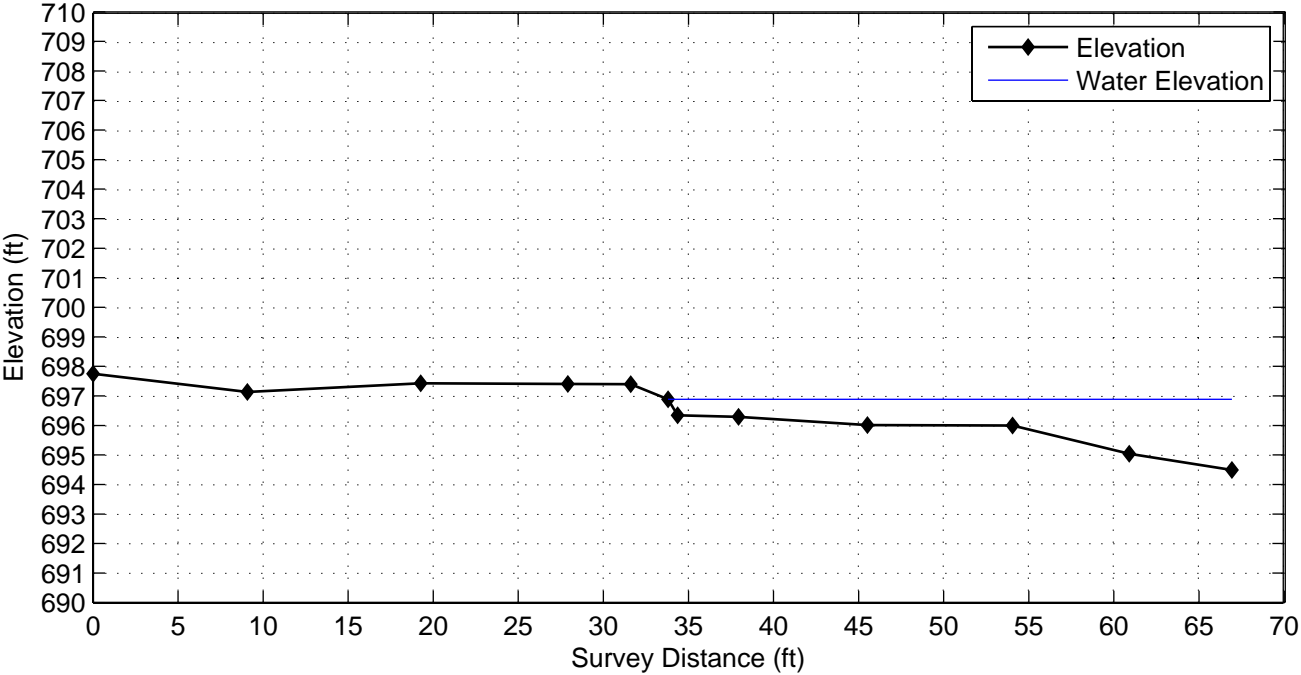
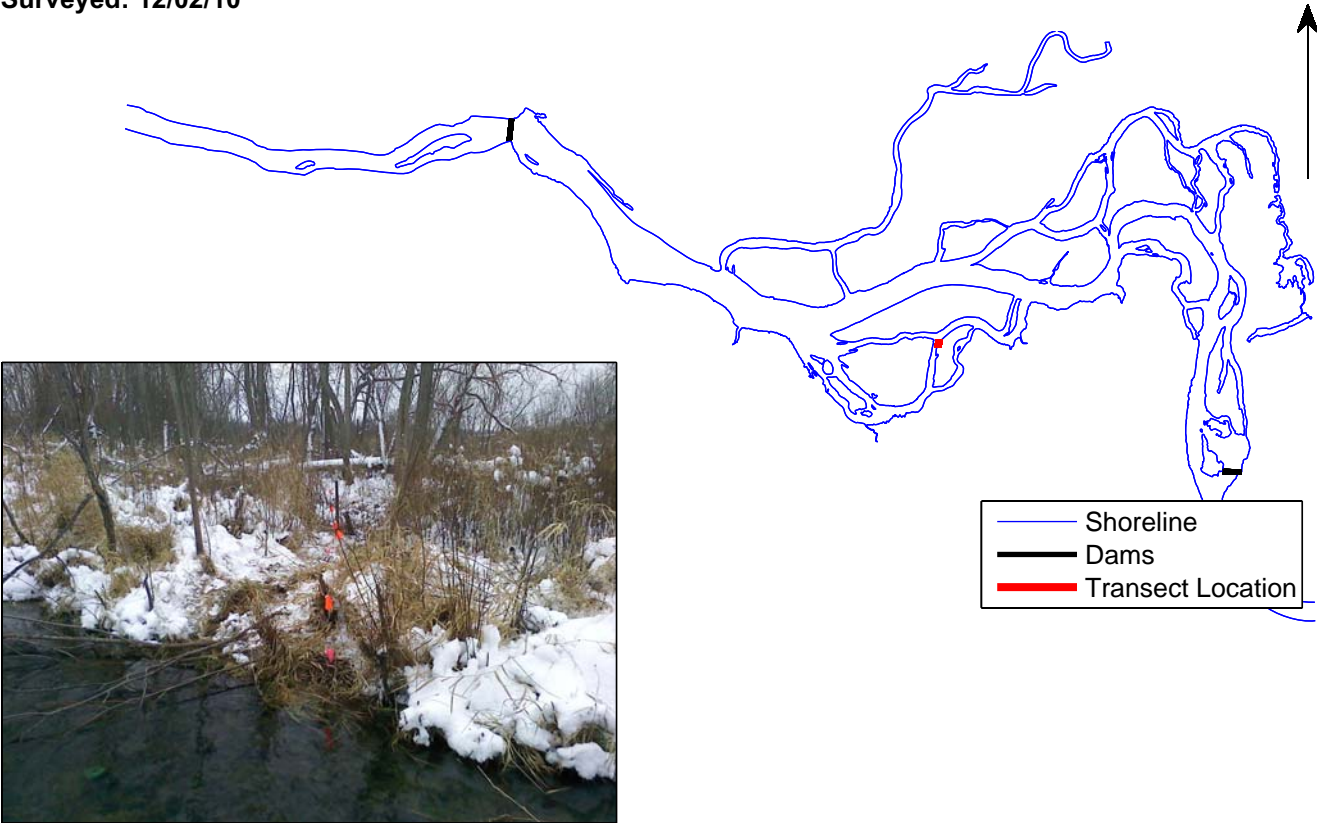
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Transect: OCBS-20
Surveyed: 12/13/10

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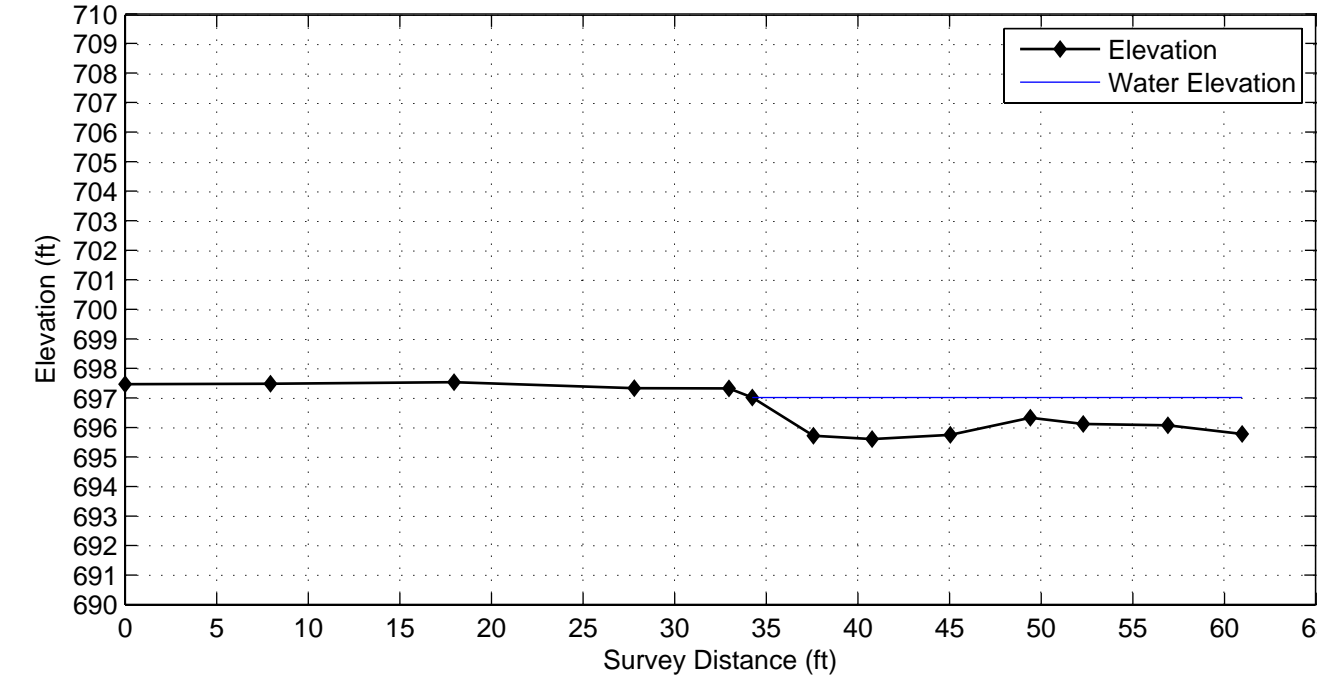
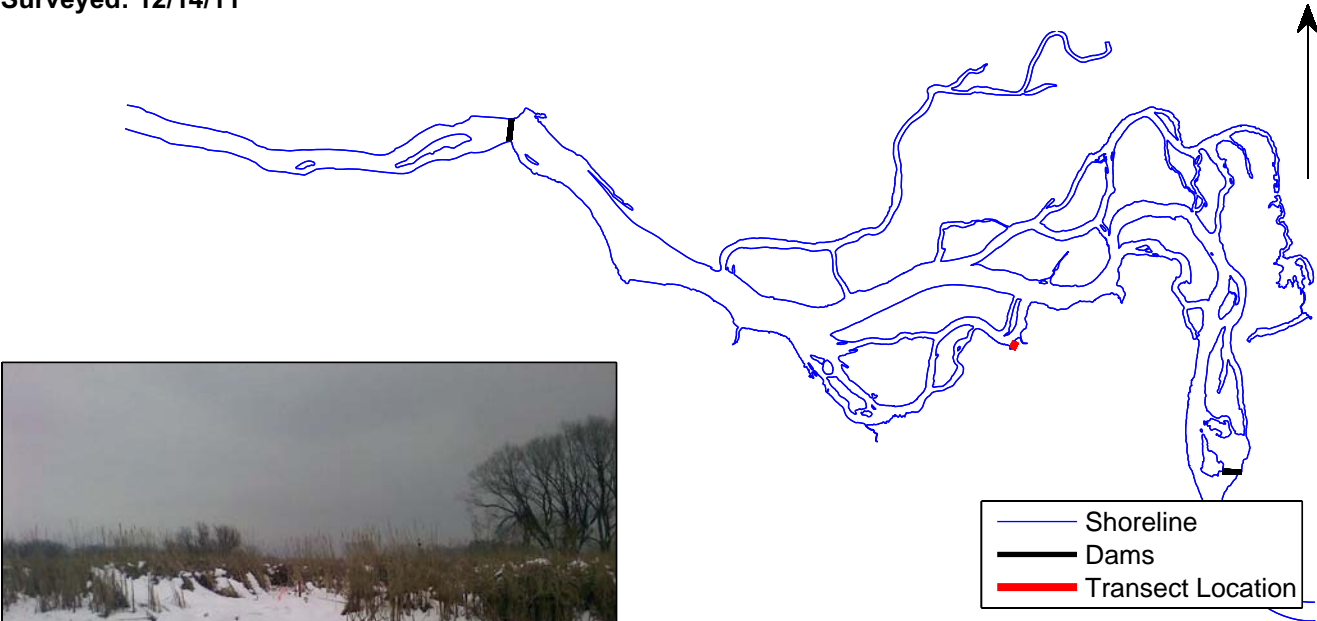
Otsego City Area 2 Bank Profiles
Transect: OCBS-21
Surveyed: 12/02/10

DRAFT



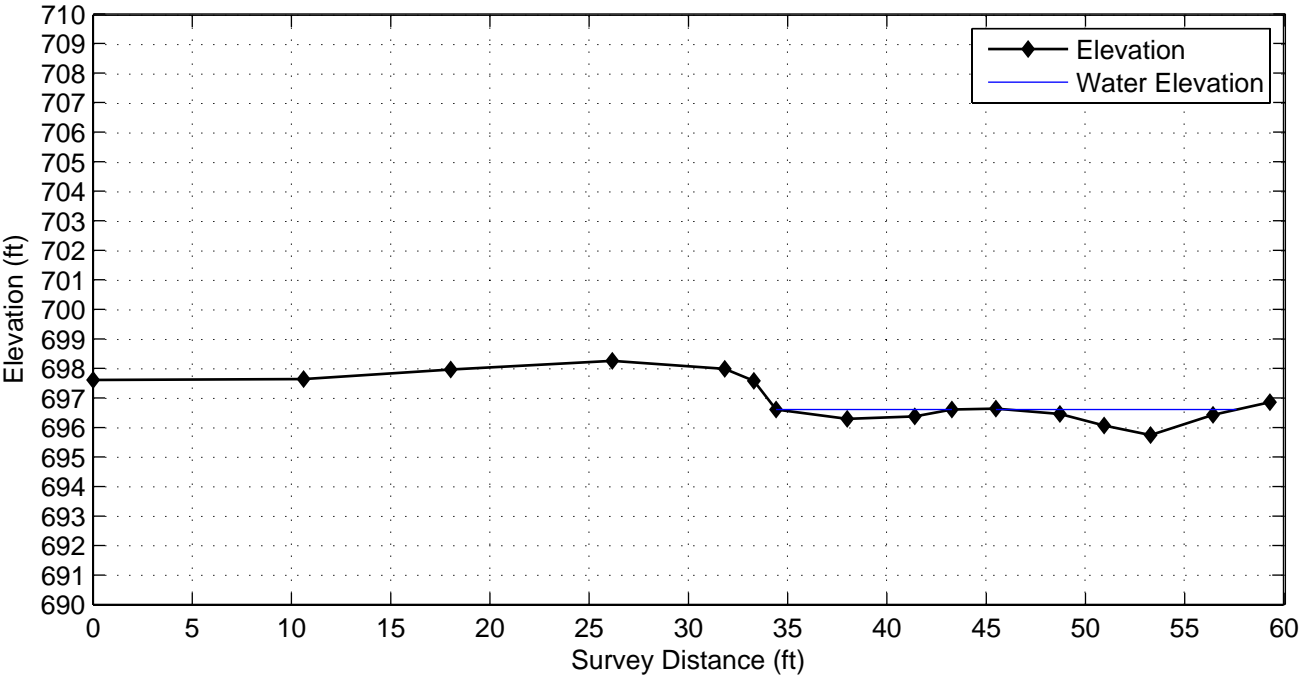
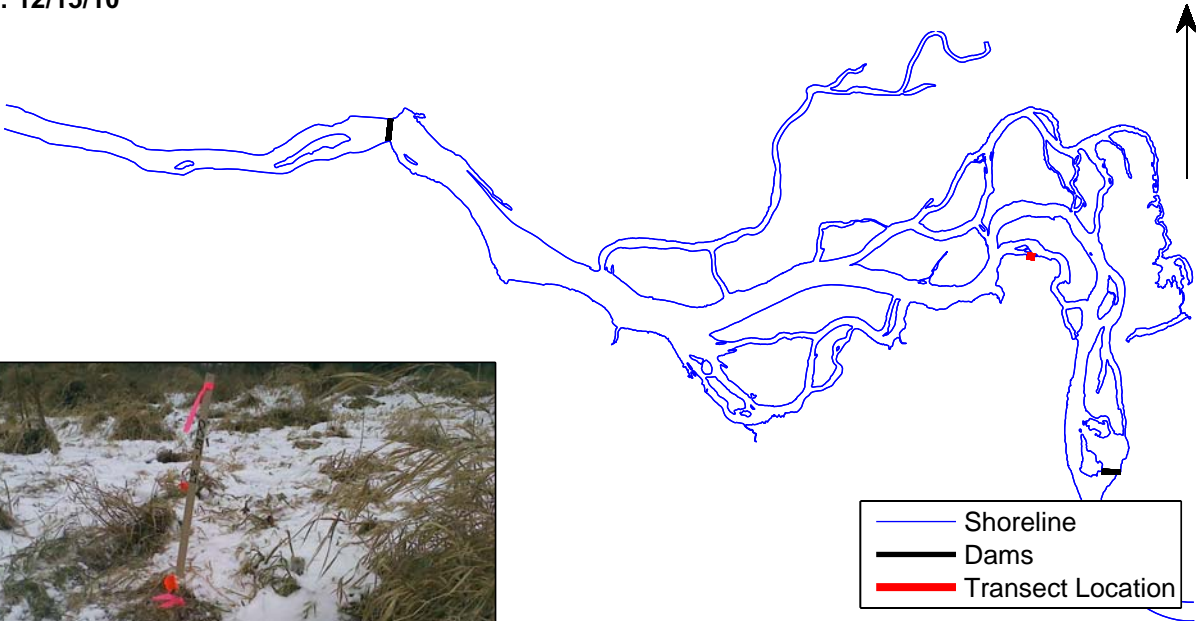
Otsego City Area 2 Bank Profiles
Transect: OCBS-22
Surveyed: 12/14/11

DRAFT



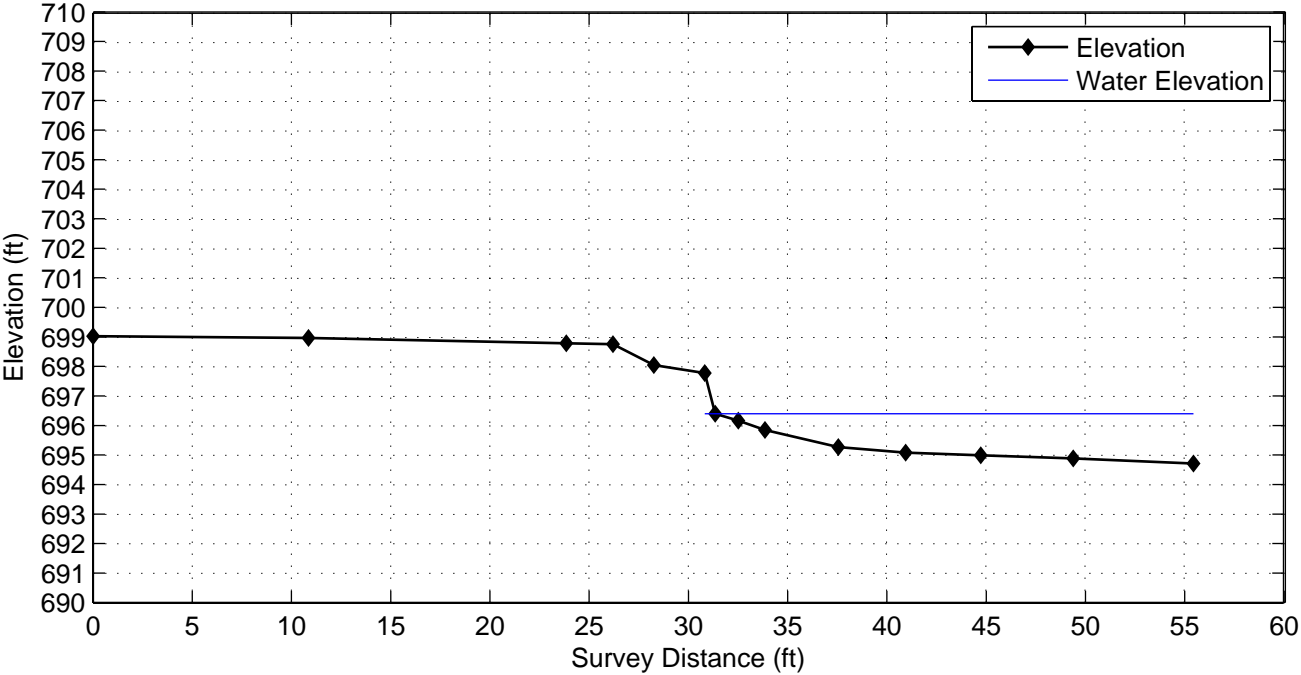
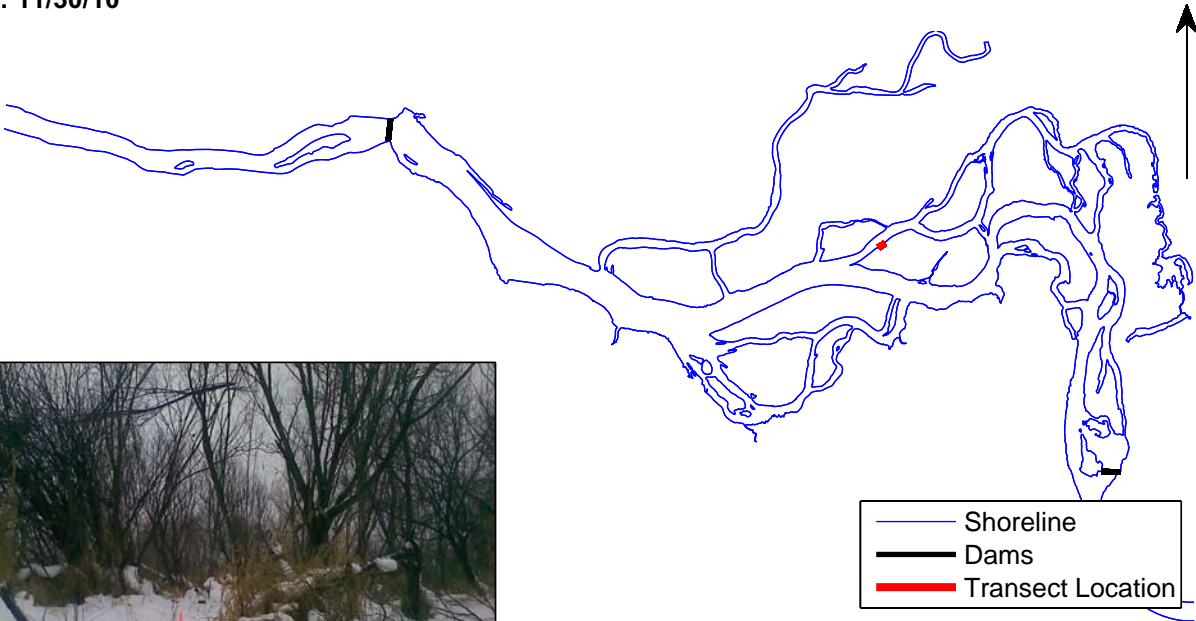
Otsego City Area 2 Bank Profiles
Transect: OCBS-23
Surveyed: 12/15/10

DRAFT



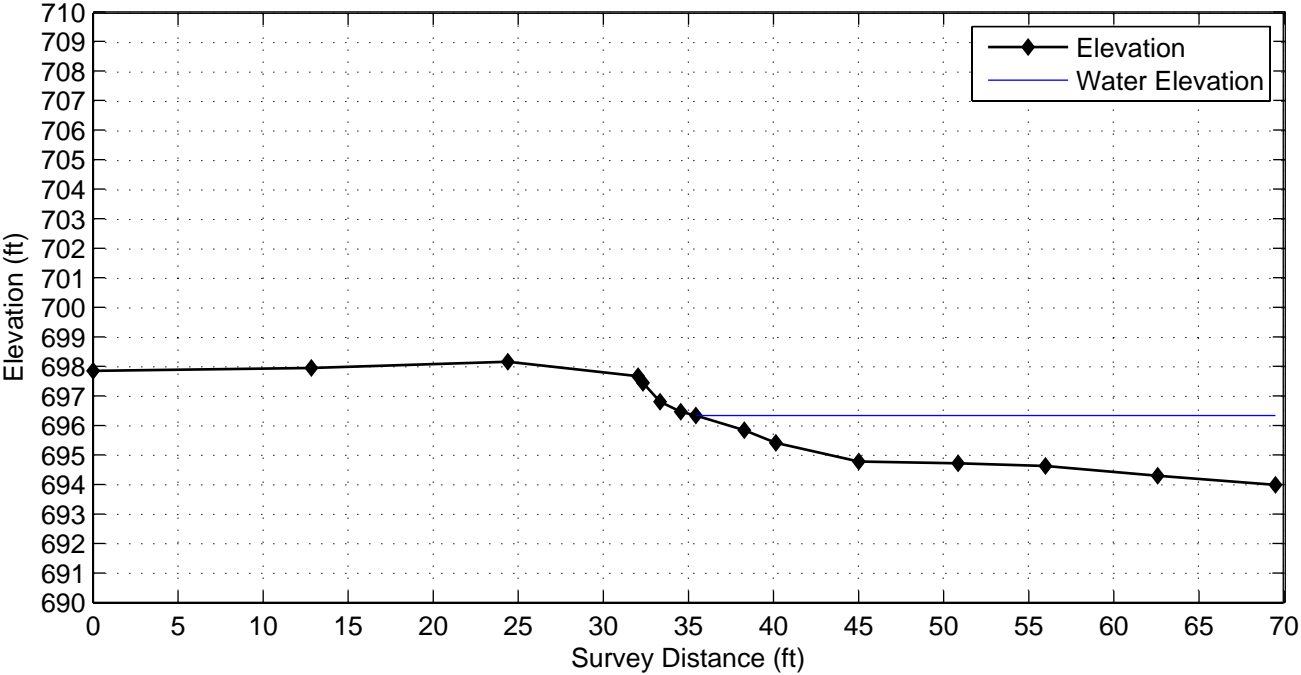
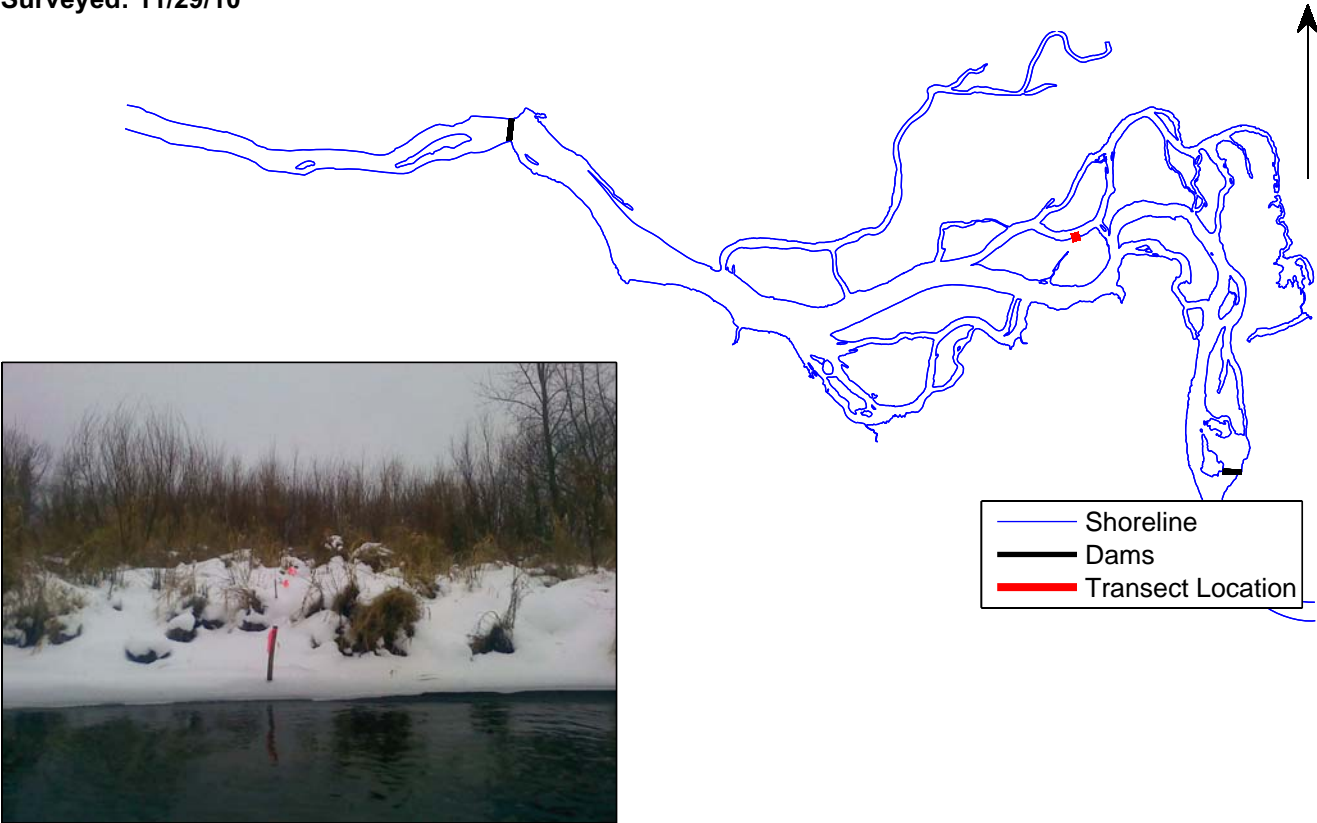
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Transect: OCBS-24
Surveyed: 11/30/10

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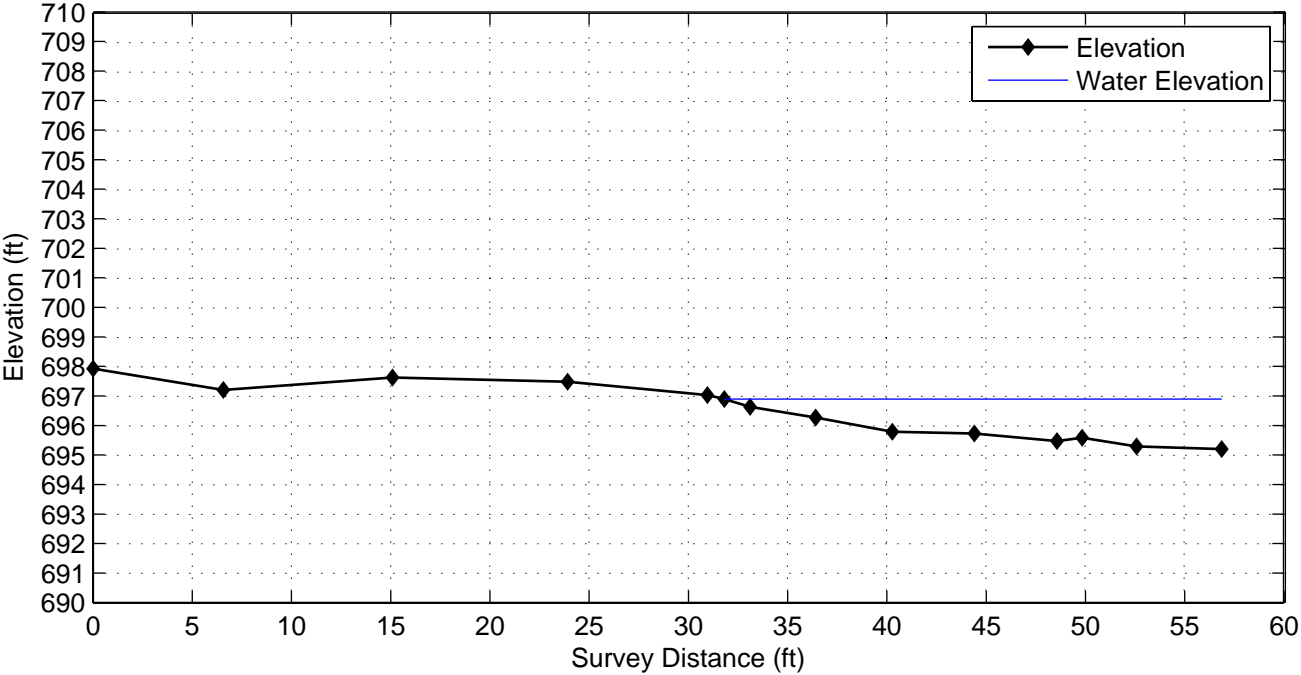
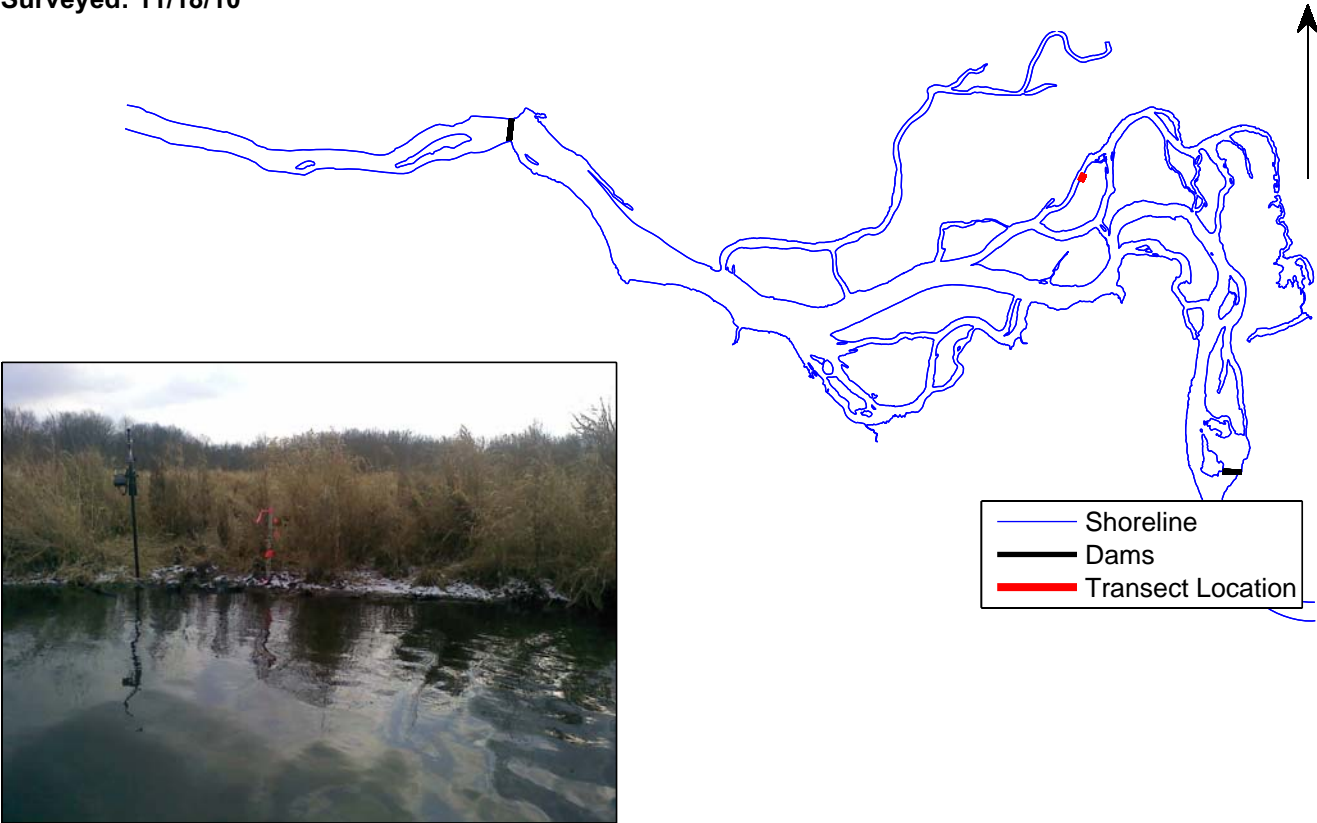
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Transect: OCBS-25
Surveyed: 11/29/10

DRAFT



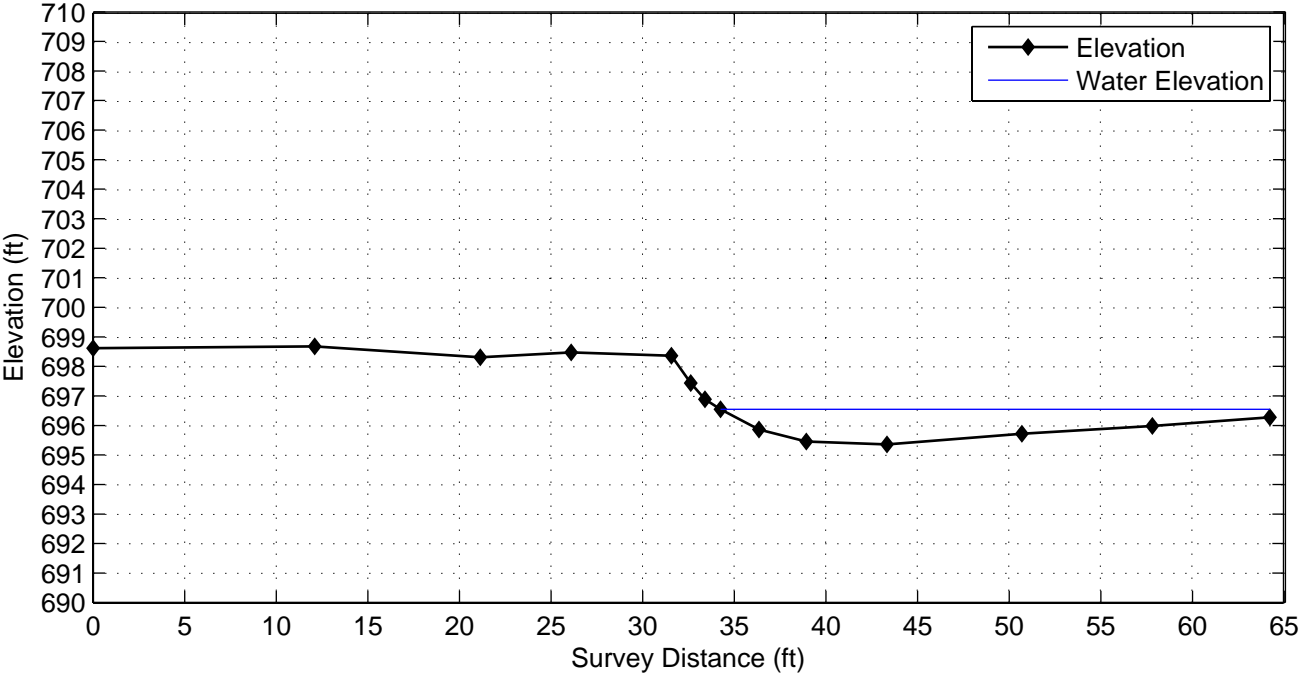
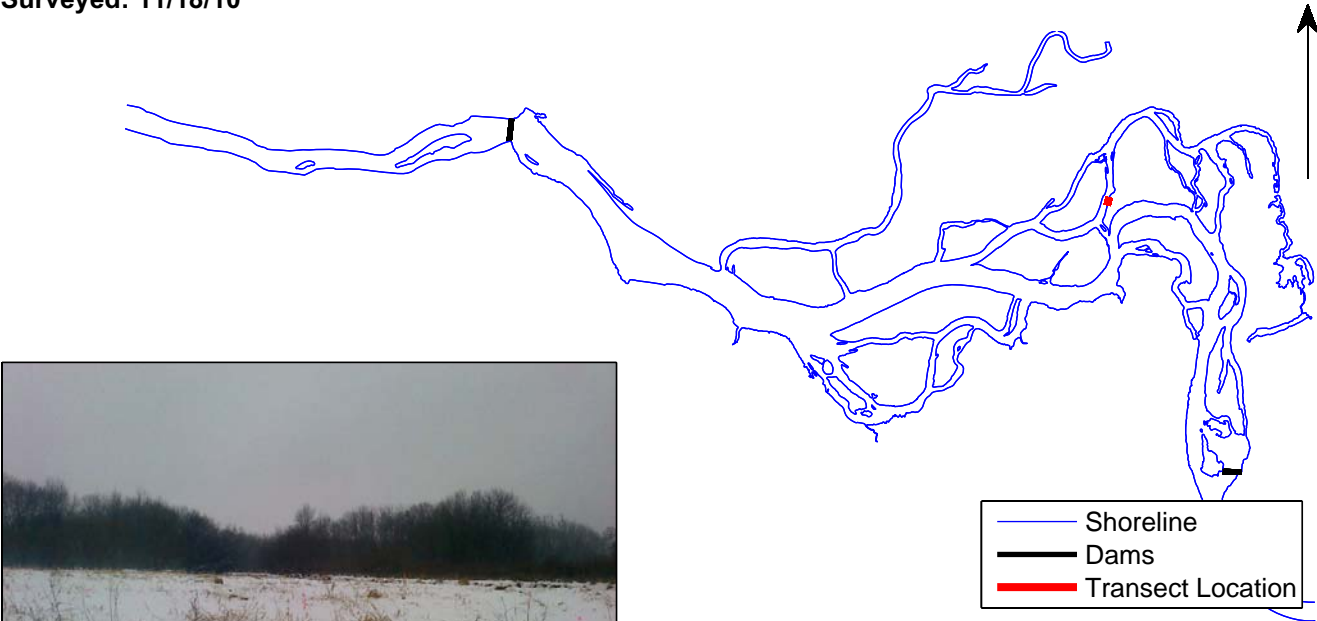
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Transect: OCBS-26
Surveyed: 11/18/10

DRAFT



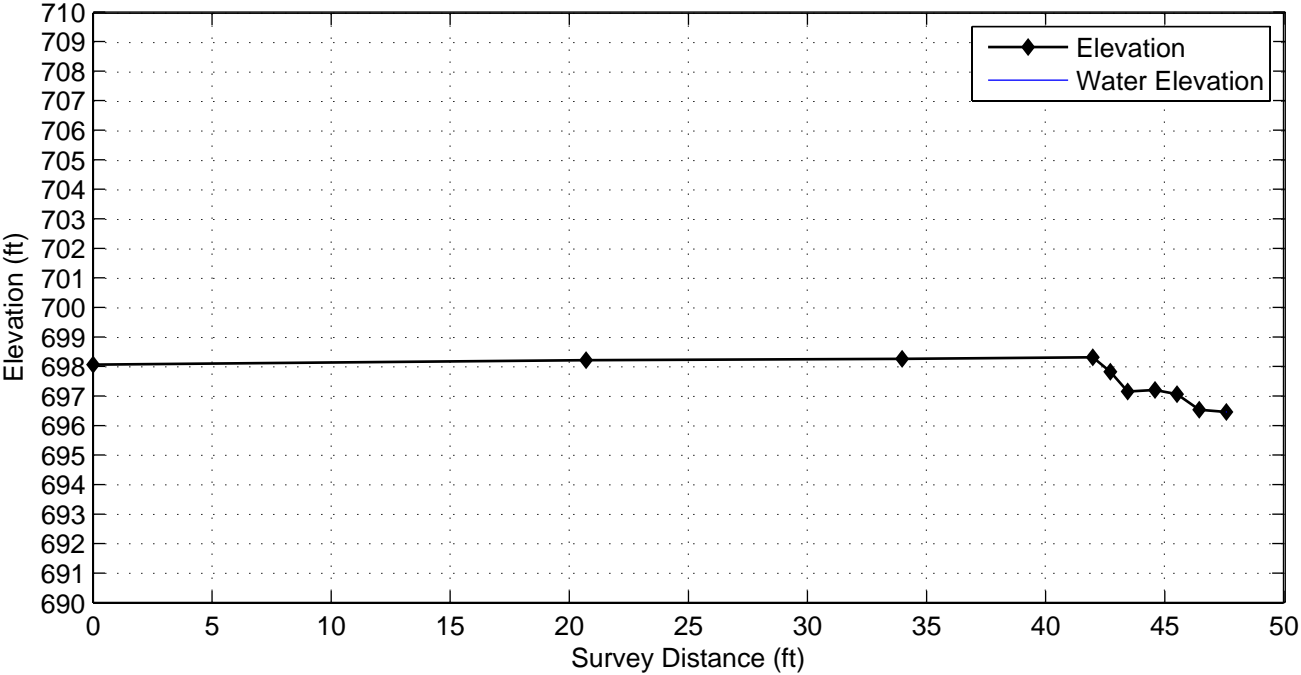
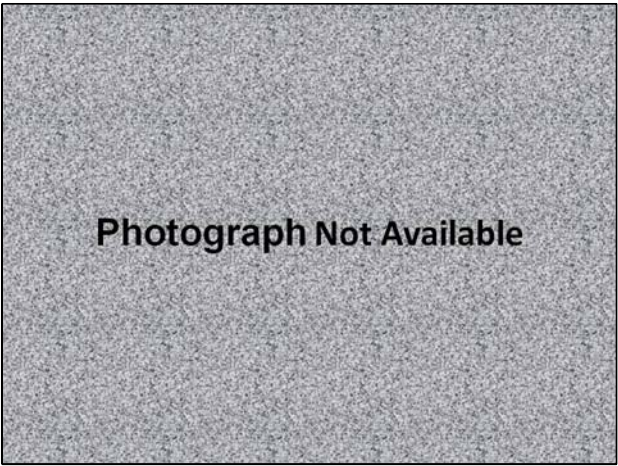
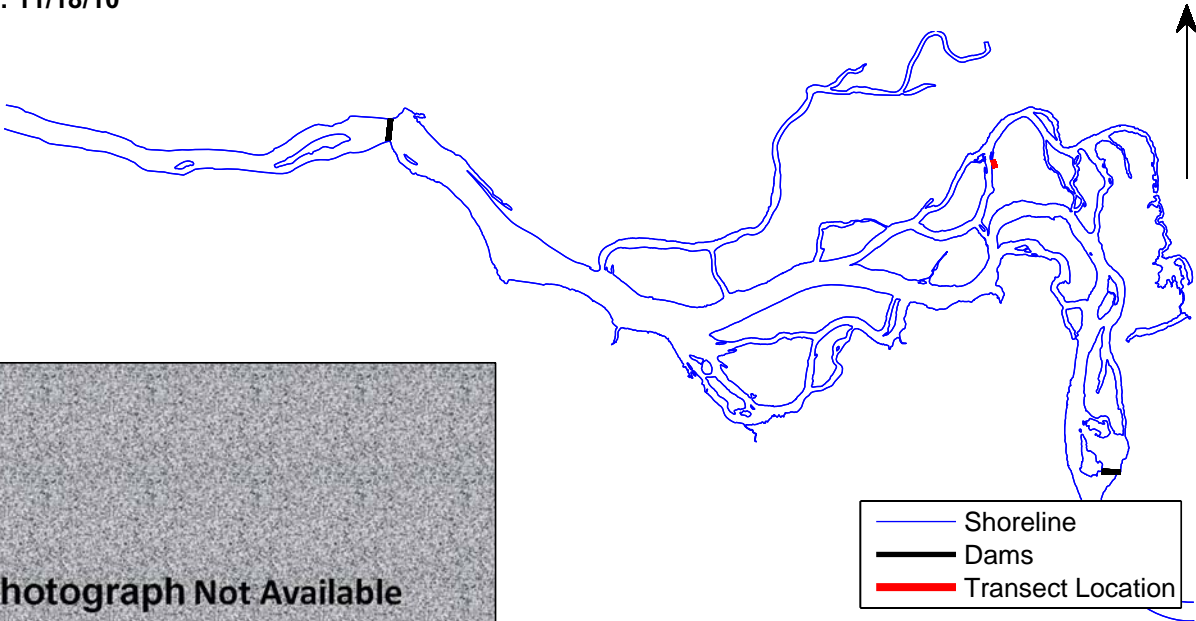
Otsego City Area 2 Bank Profiles
Transect: OCBS-27
Surveyed: 11/18/10

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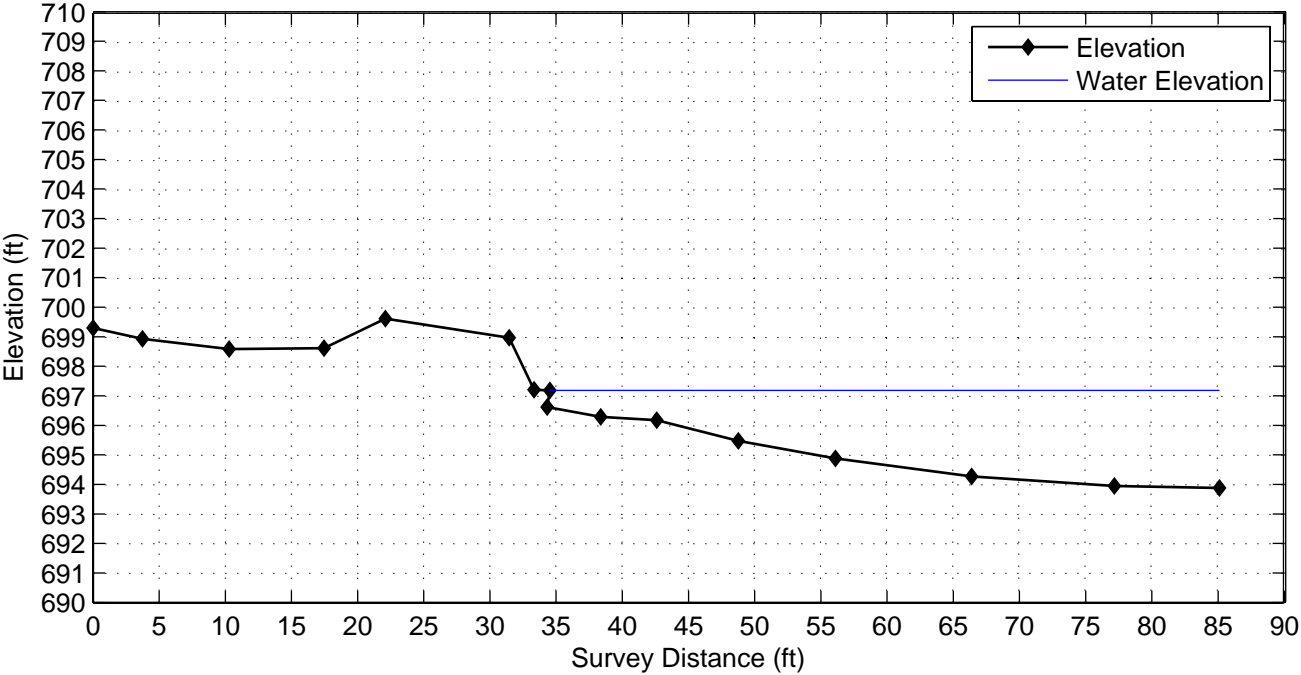
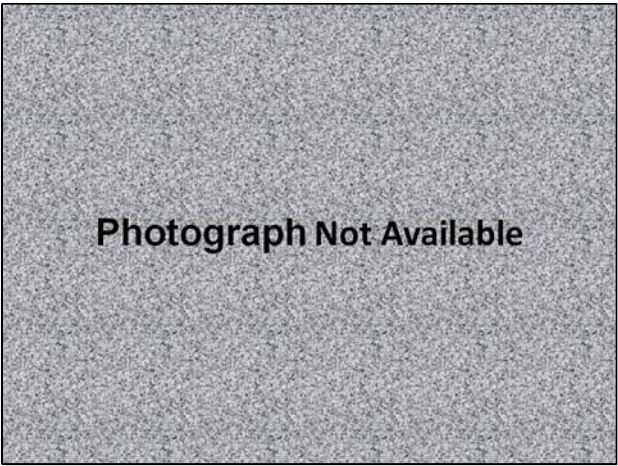
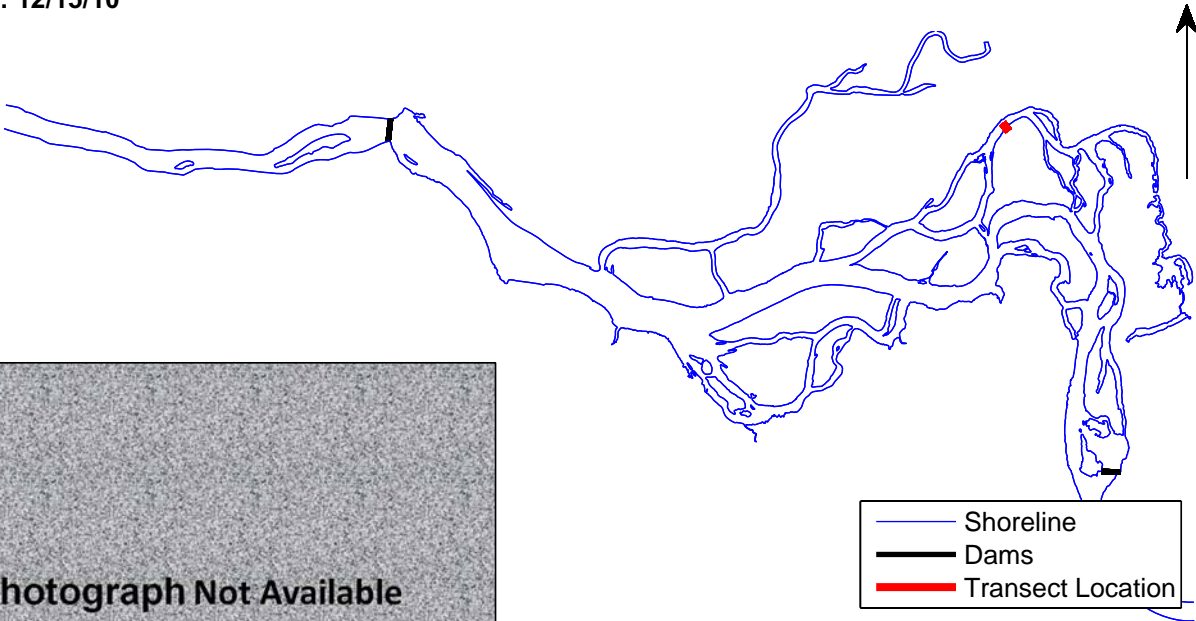
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Transect: OCBS-28
Surveyed: 11/18/10

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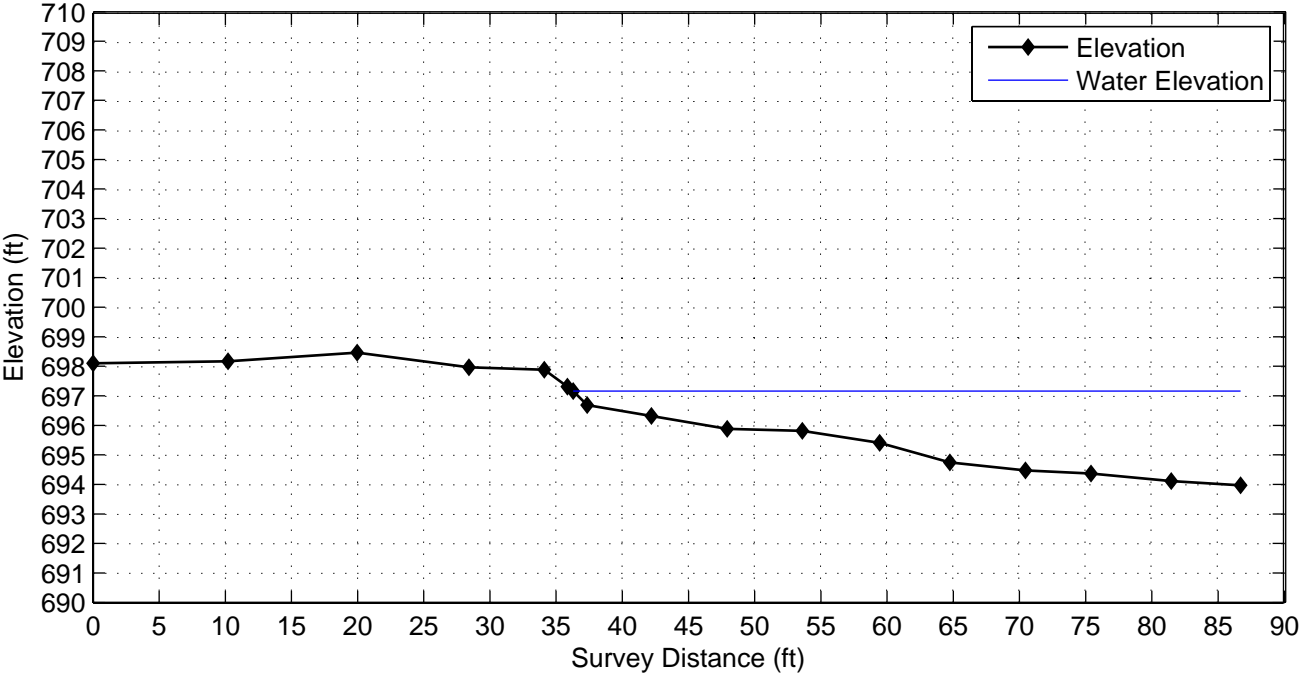
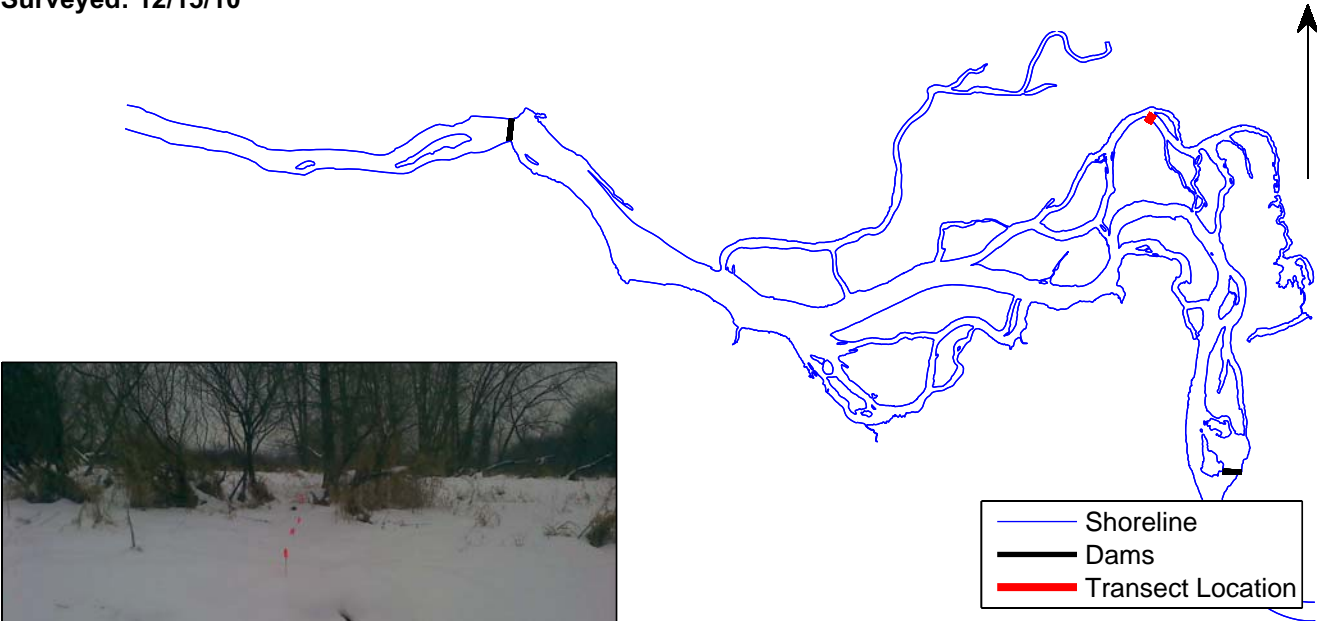
Otsego City Area 2 Bank Profiles
Transect: OCBS-29
Surveyed: 12/15/10

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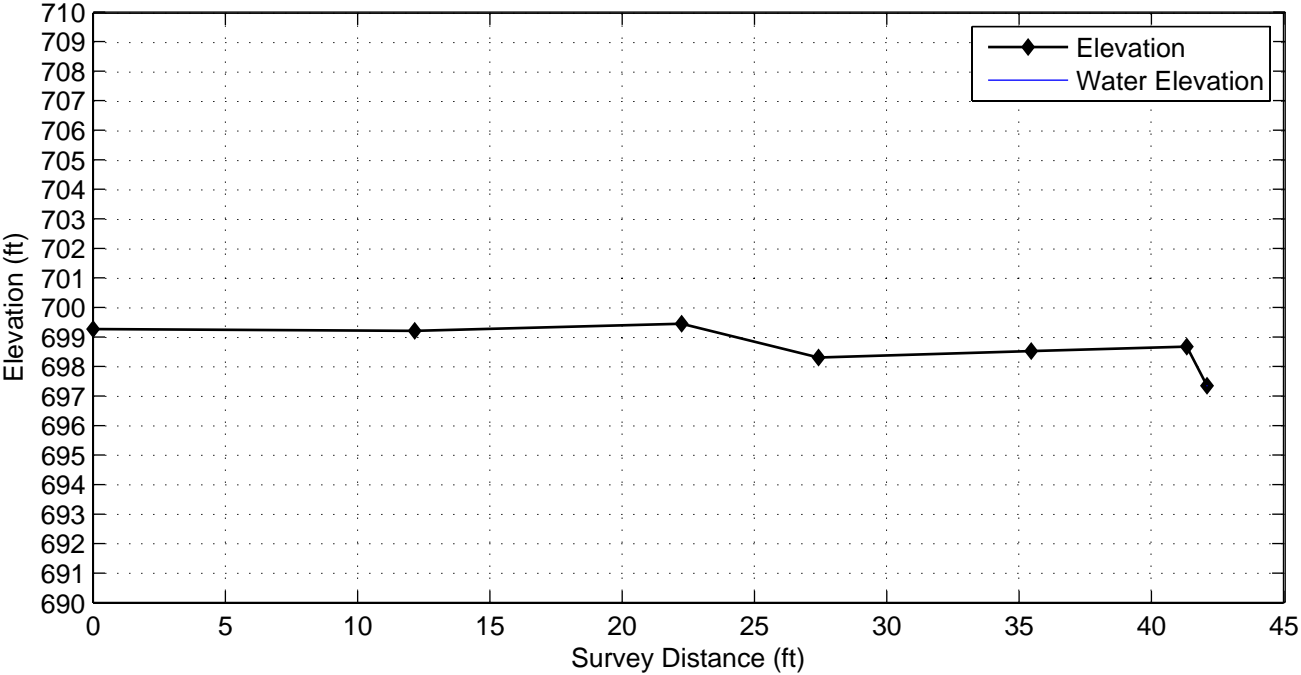
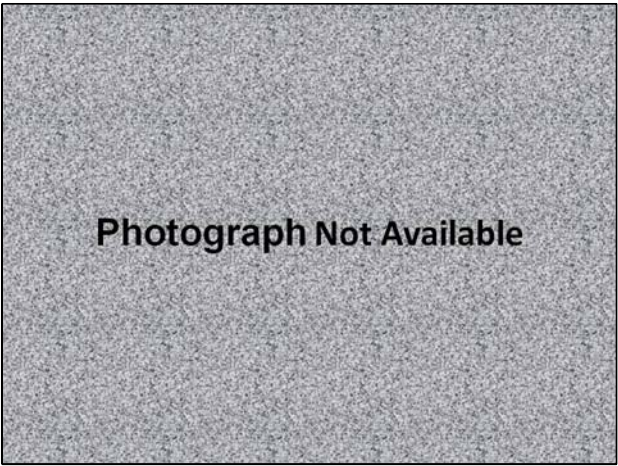
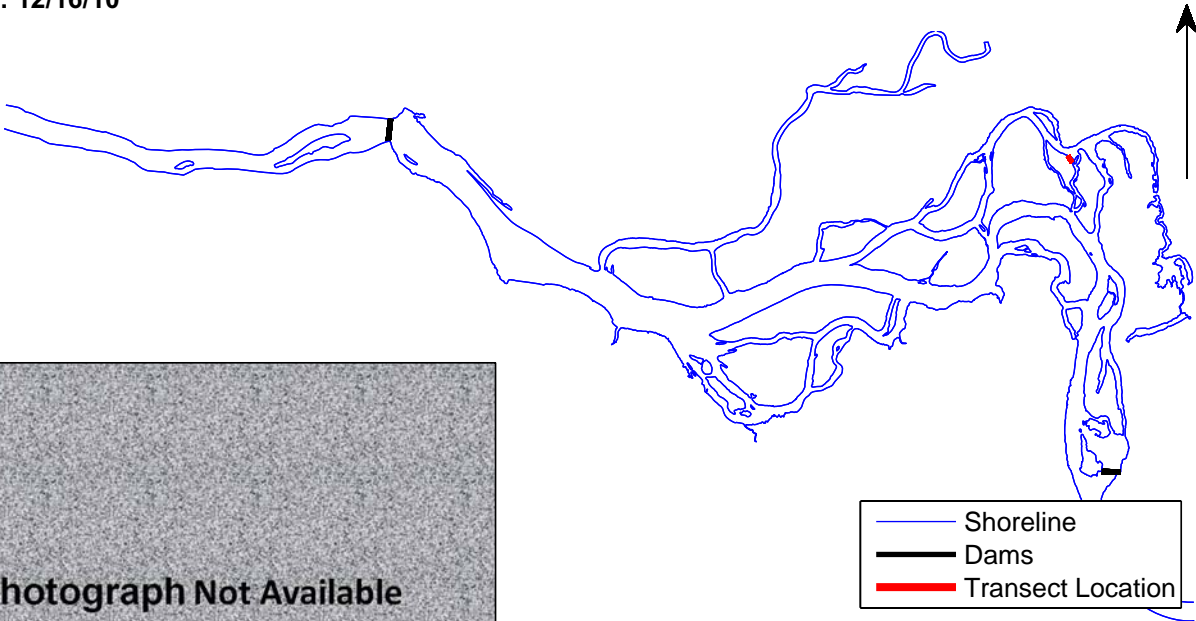
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Transect: OCBS-30
Surveyed: 12/15/10

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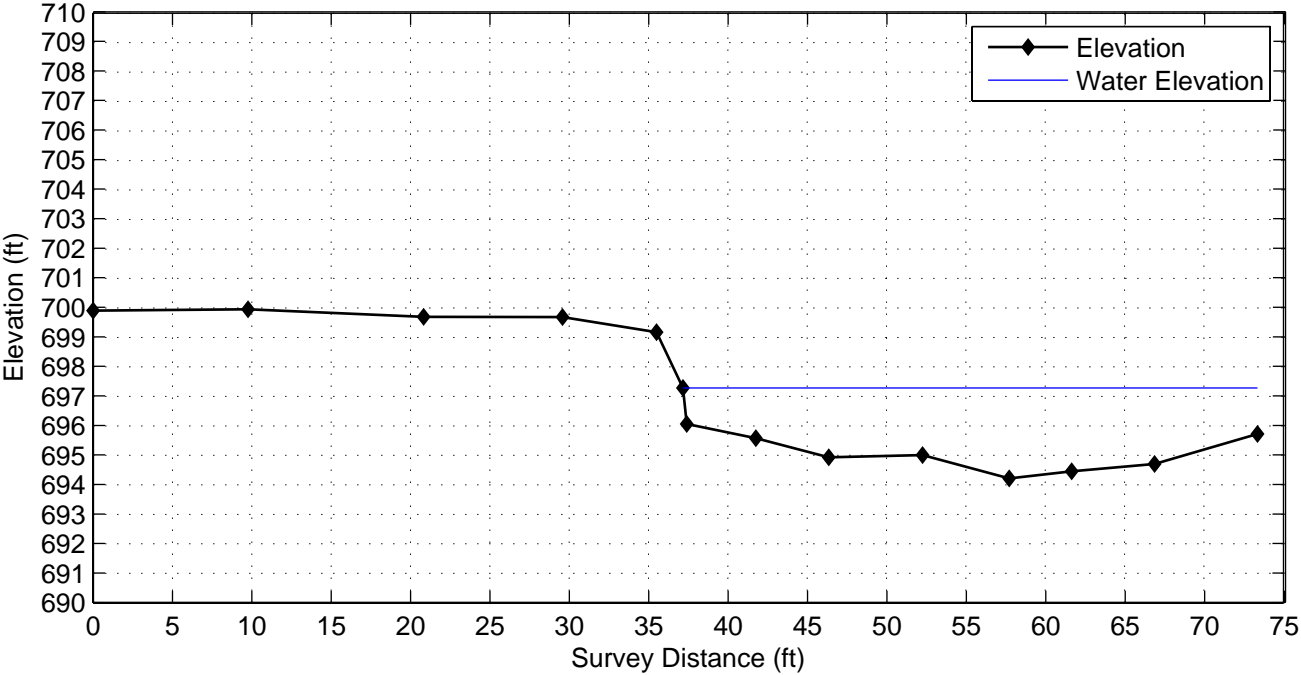
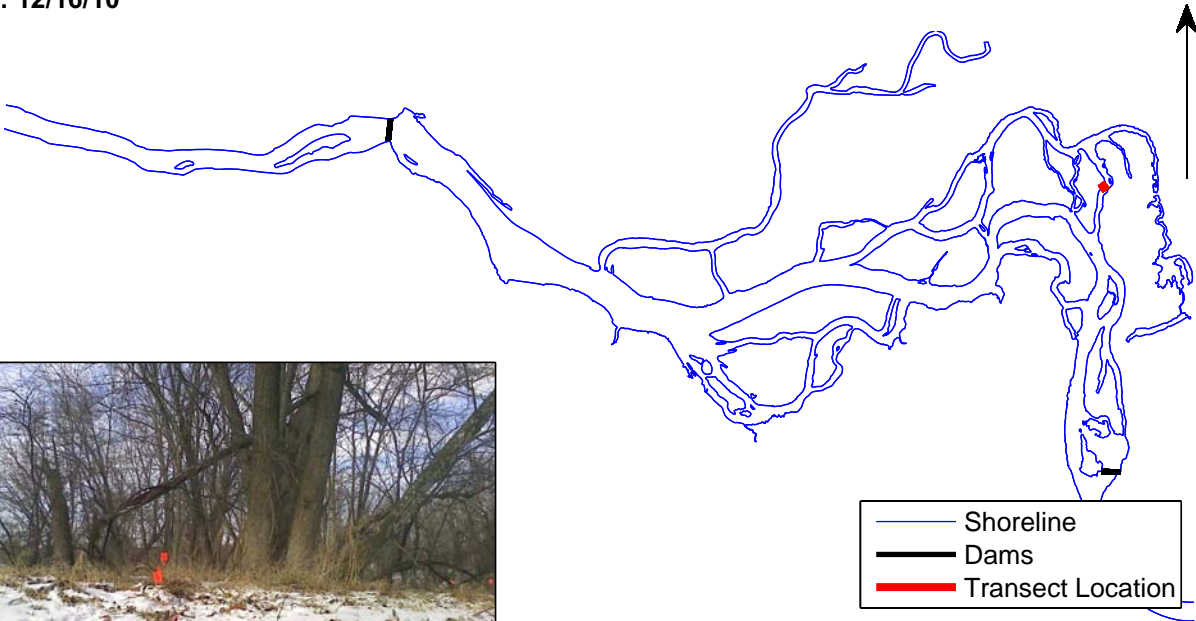
Otsego City Area 2 Bank Profiles
Transect: OCBS-31
Surveyed: 12/16/10

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Otsego City Area 2 Bank Profiles
Transect: OCBS-32
Surveyed: 12/16/10

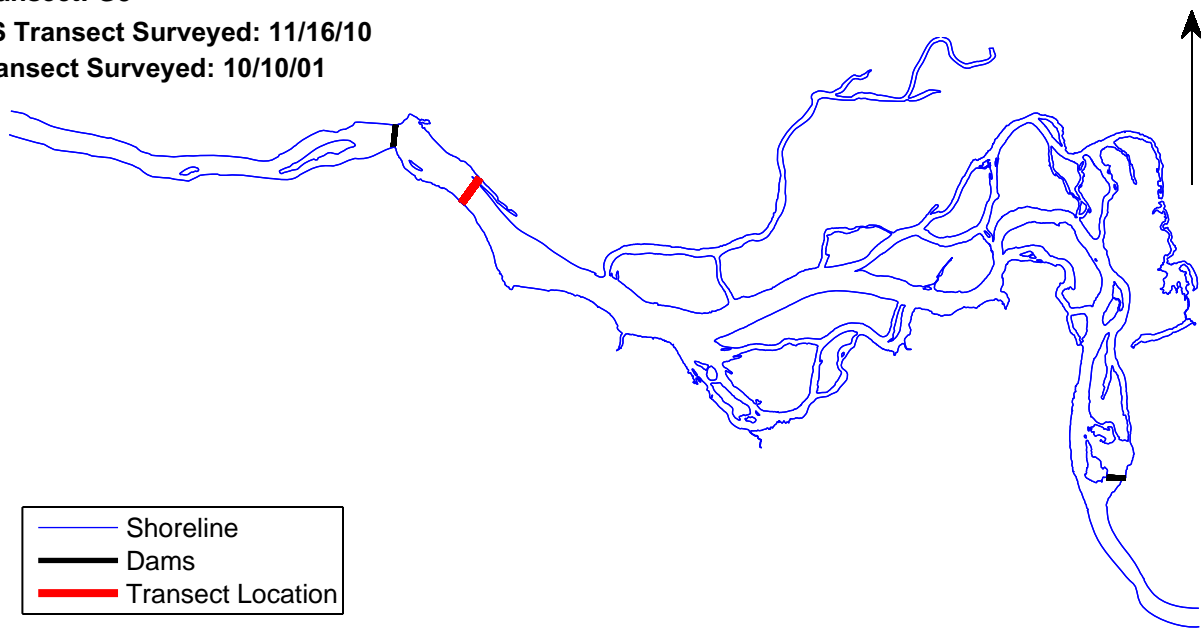
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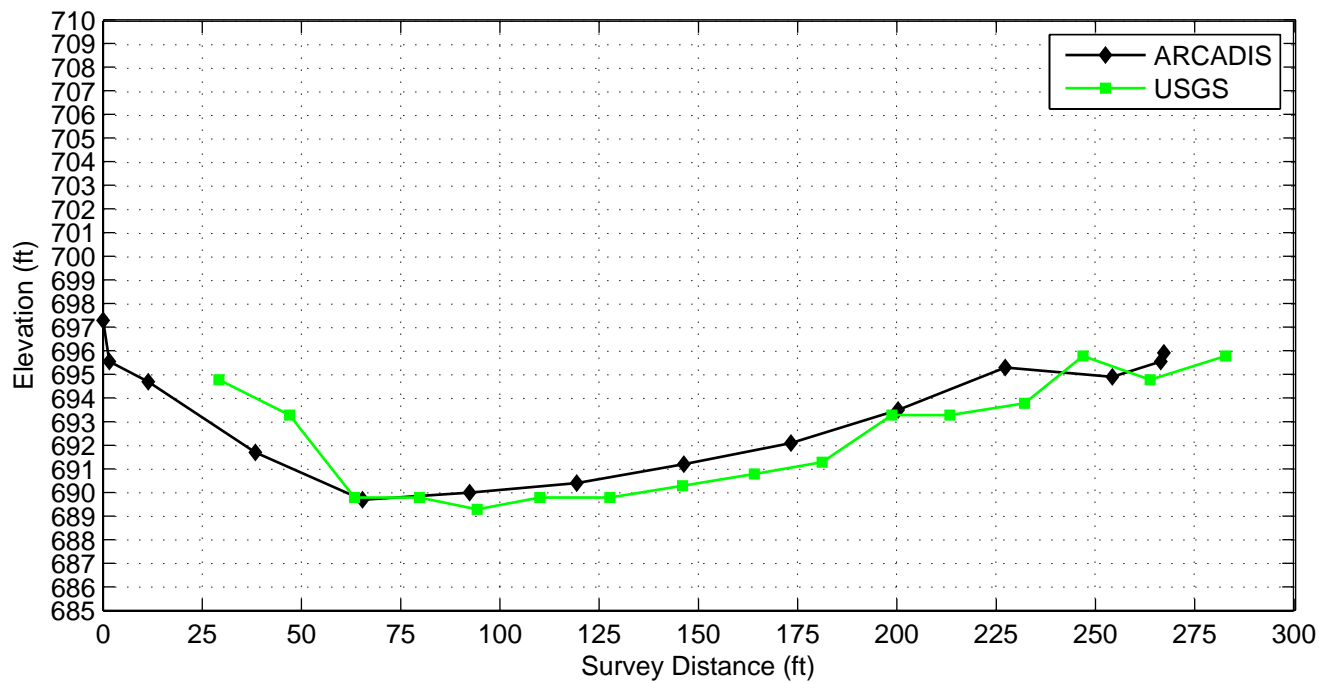
Transect Comparisons

Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-02
USGS Transect: G3
ARCADIS Transect Surveyed: 11/16/10
USGS Transect Surveyed: 10/10/01

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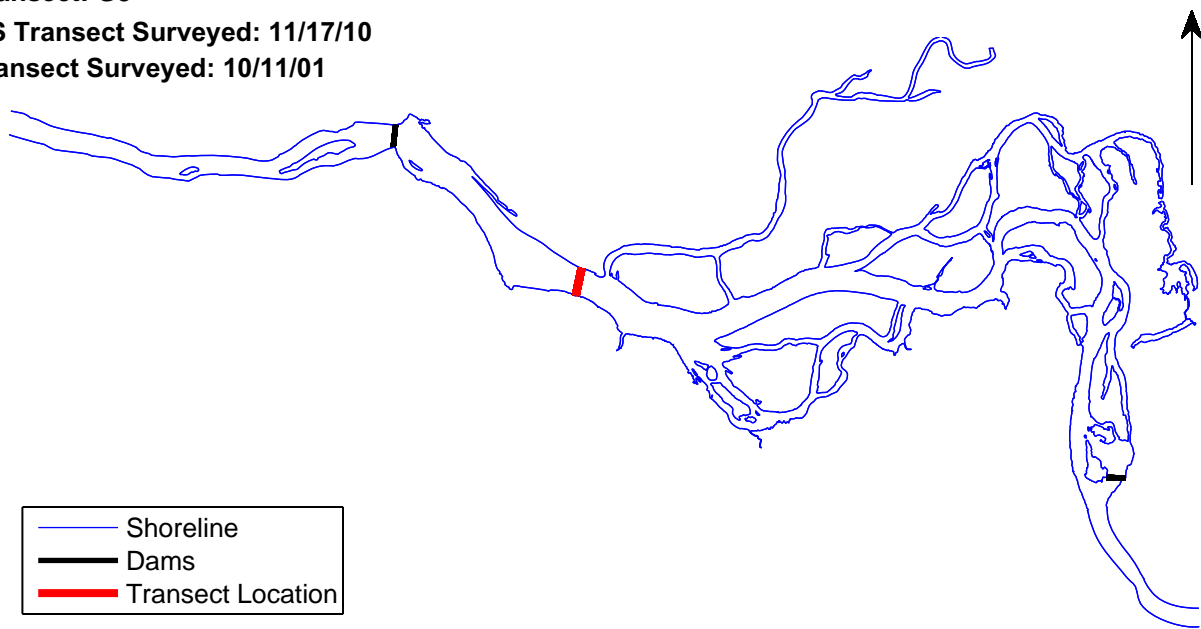


Orientation Looking Downstream

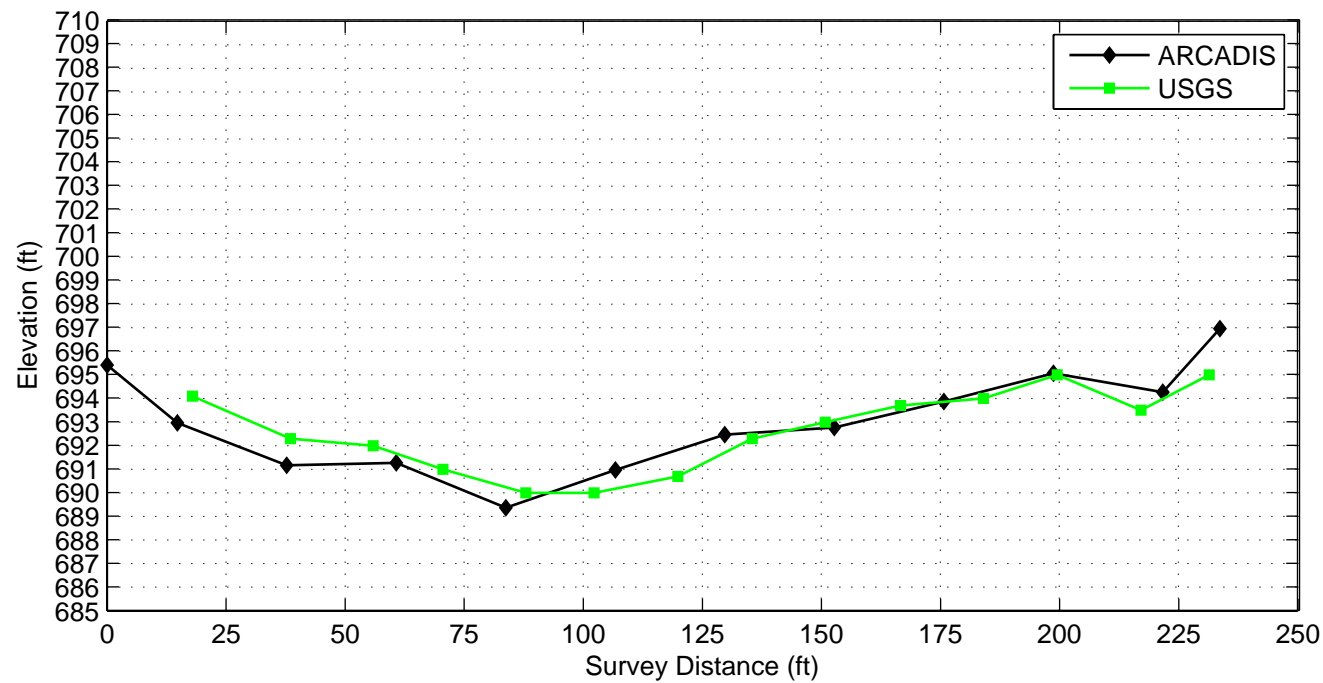


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-04
USGS Transect: G5
ARCADIS Transect Surveyed: 11/17/10
USGS Transect Surveyed: 10/11/01

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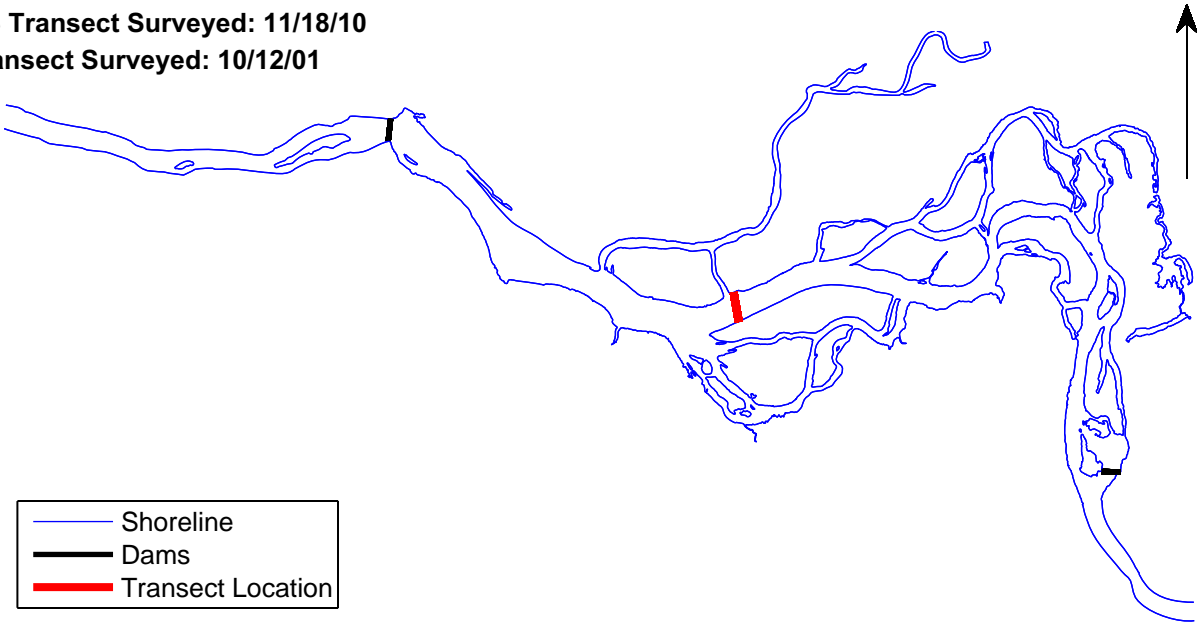


Orientation Looking Downstream

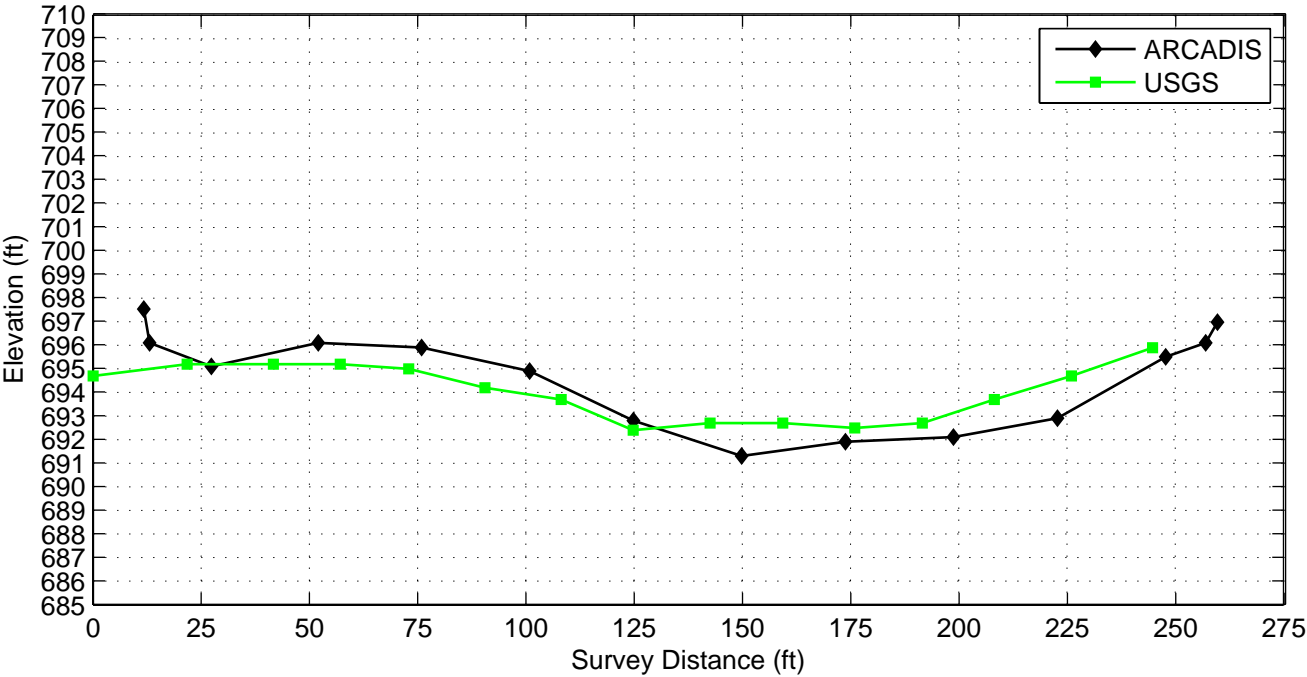


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-07
USGS Transect: G7
ARCADIS Transect Surveyed: 11/18/10
USGS Transect Surveyed: 10/12/01

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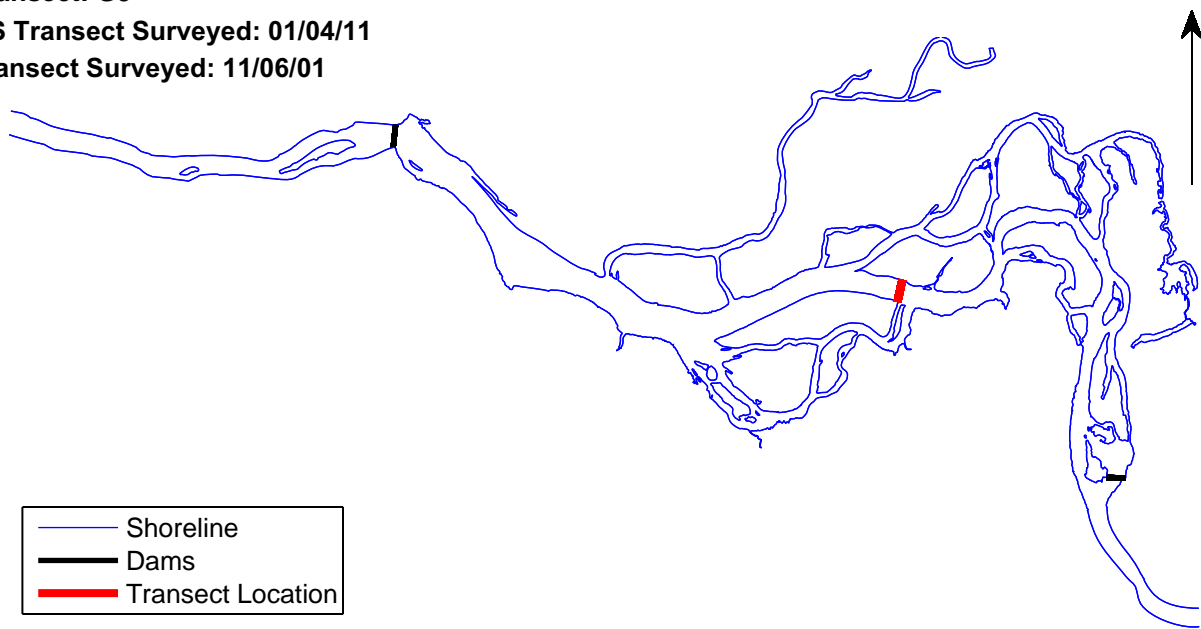


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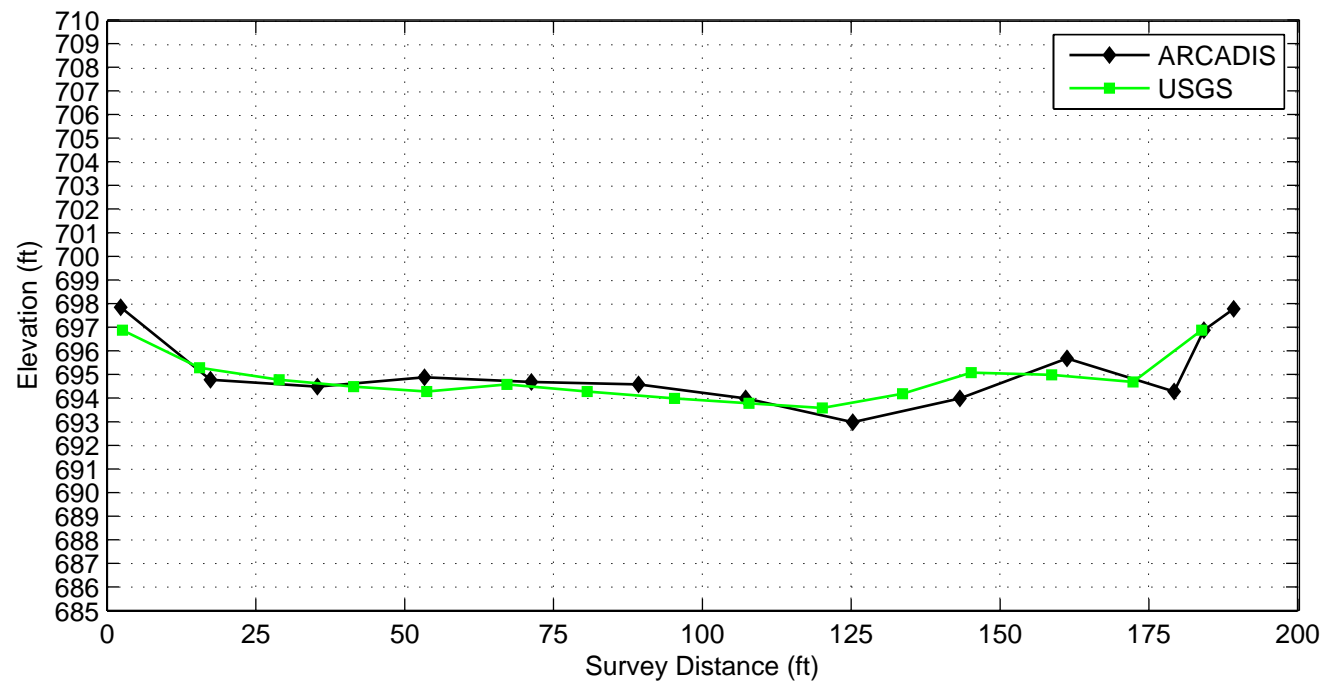


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-10
USGS Transect: G9
ARCADIS Transect Surveyed: 01/04/11
USGS Transect Surveyed: 11/06/01

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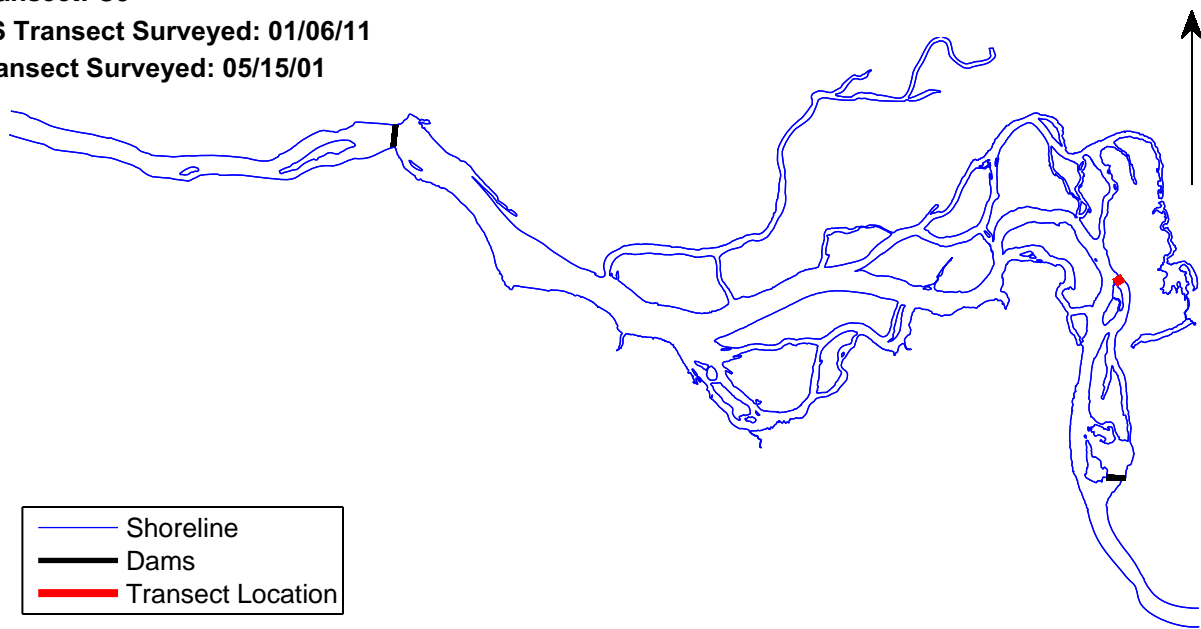


Orientation Looking Downstream

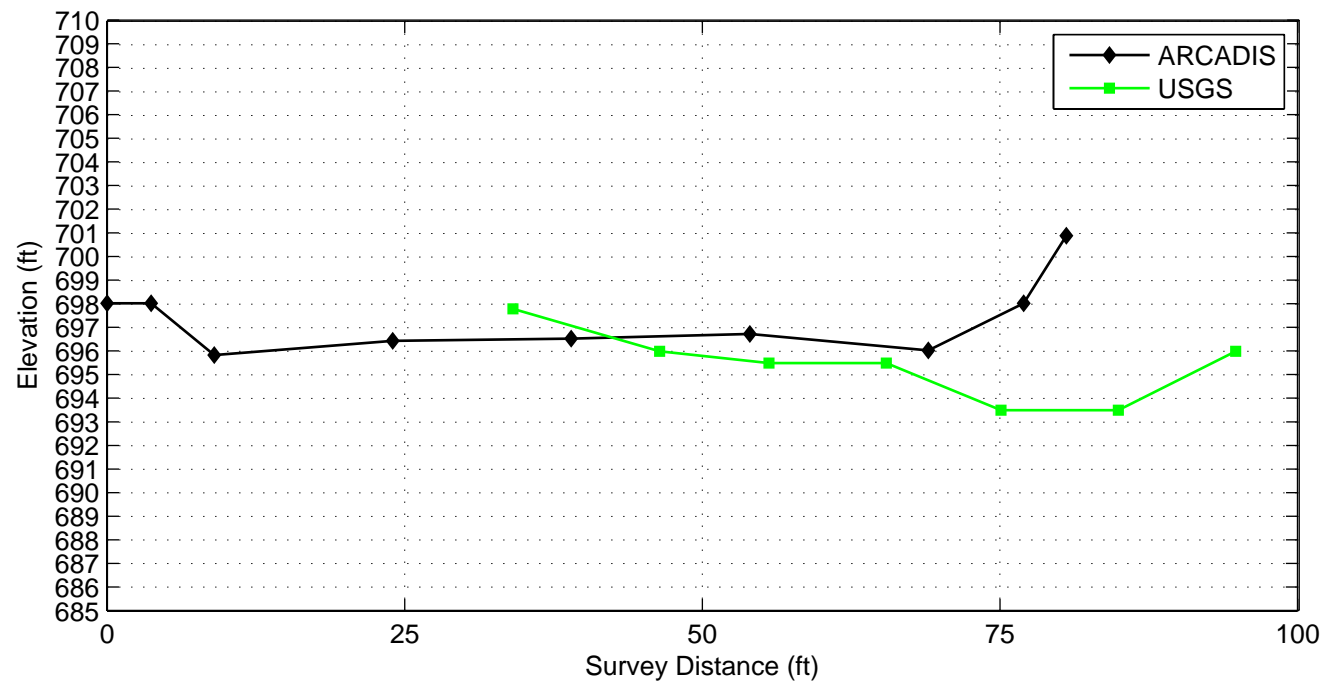


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-17
USGS Transect: C9
ARCADIS Transect Surveyed: 01/06/11
USGS Transect Surveyed: 05/15/01

DRAFT

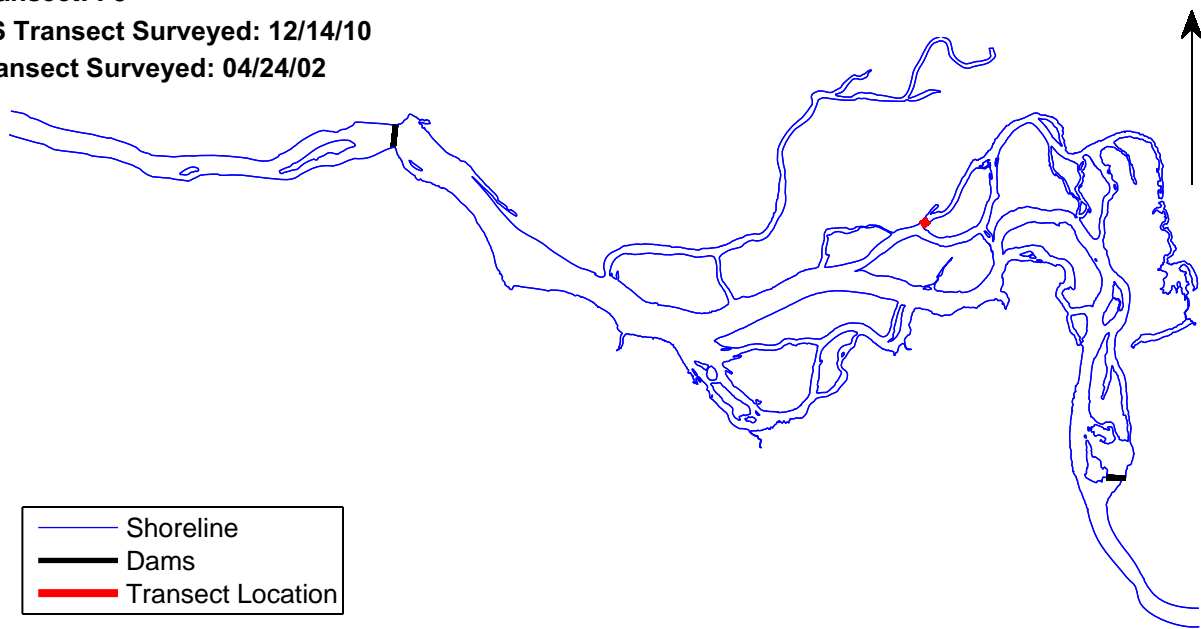


Orientation Looking Downstream

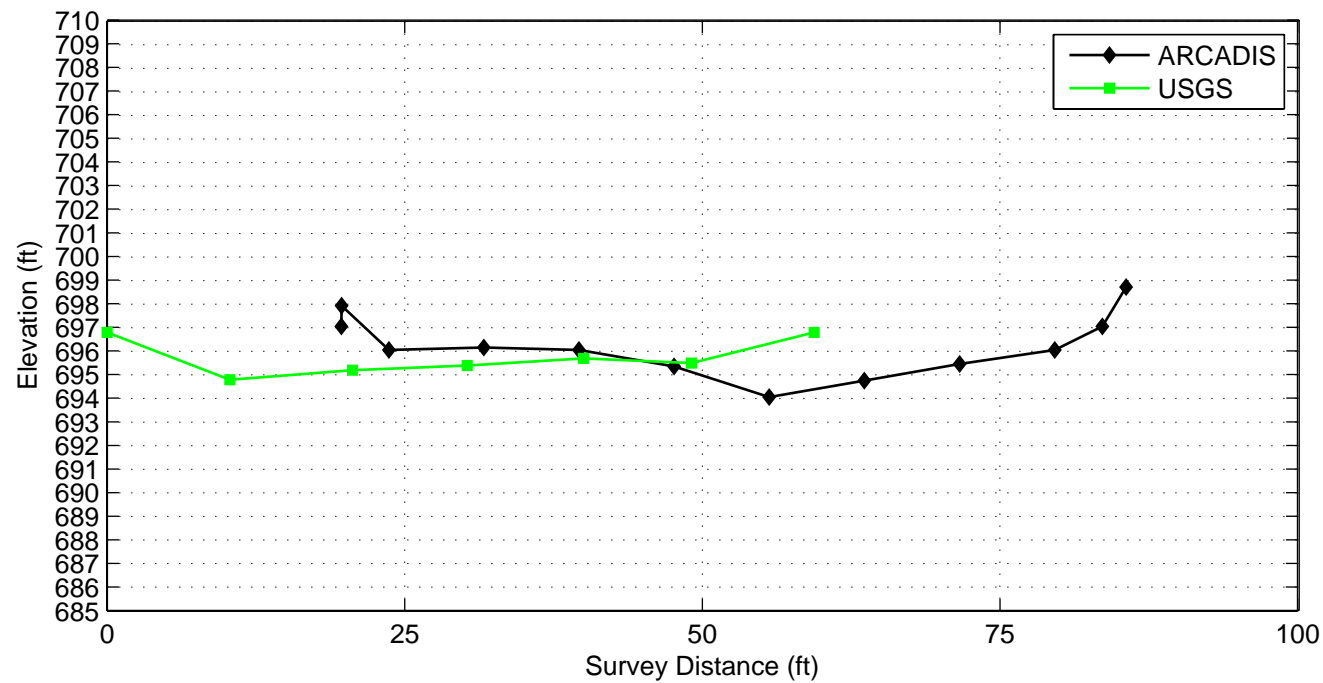


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-31
USGS Transect: F5
ARCADIS Transect Surveyed: 12/14/10
USGS Transect Surveyed: 04/24/02

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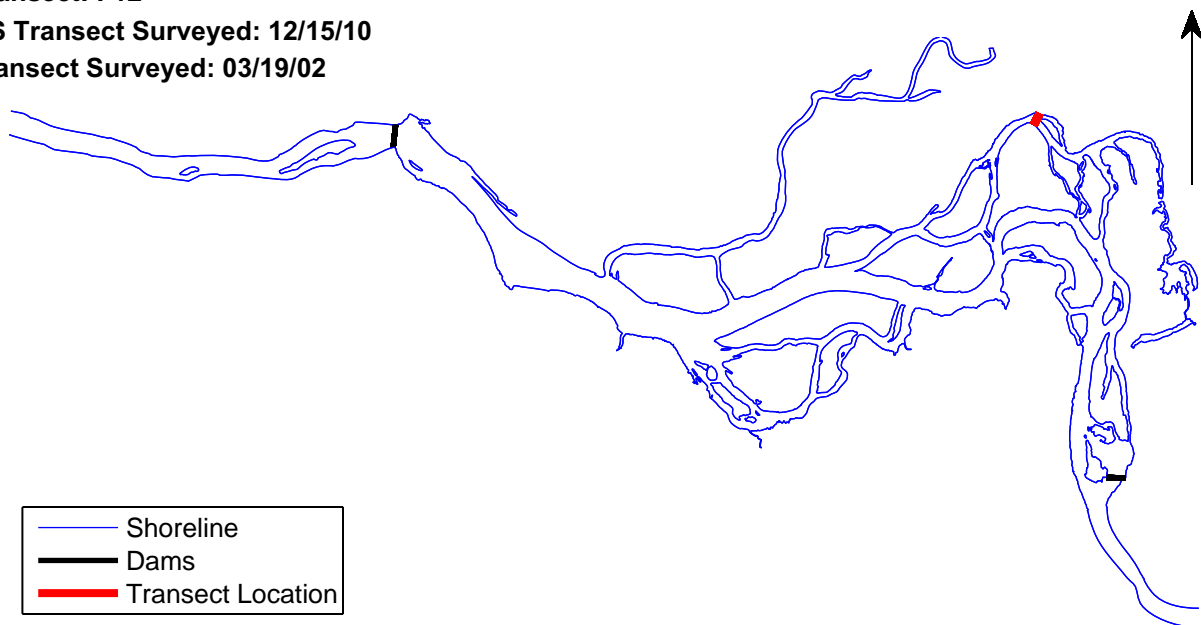


Orientation Looking Downstream

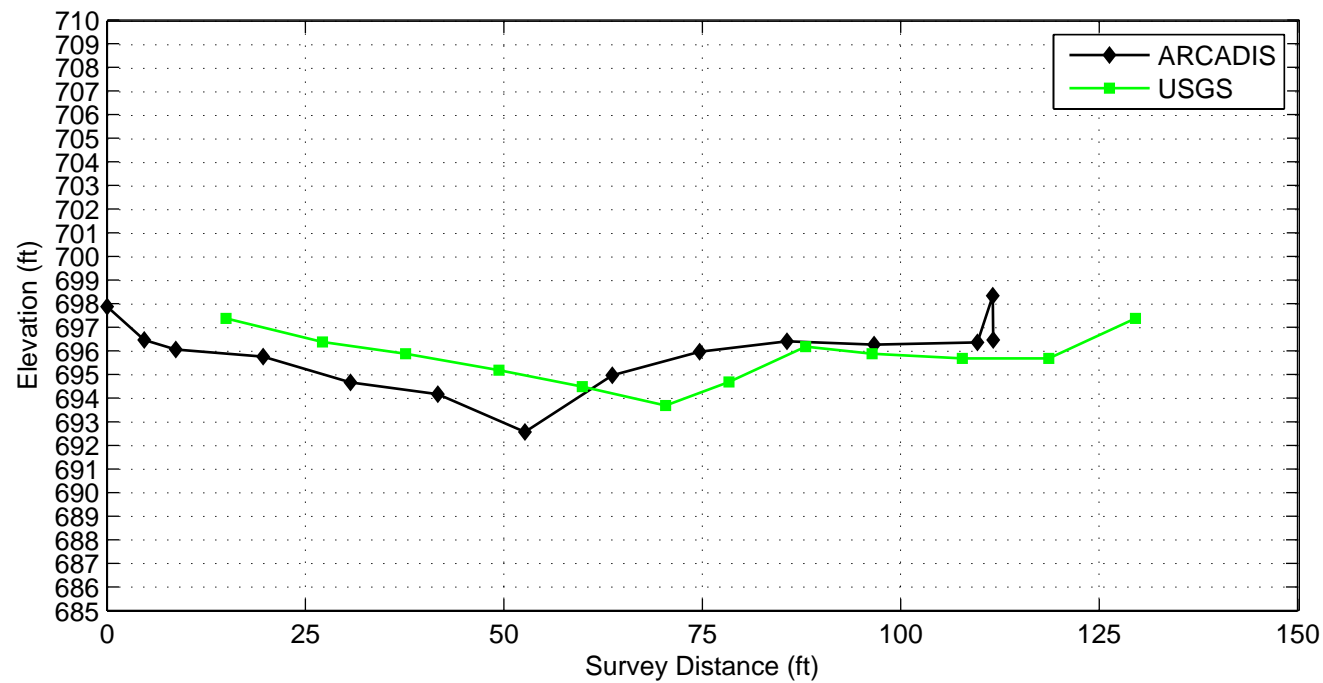


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-36
USGS Transect: F12
ARCADIS Transect Surveyed: 12/15/10
USGS Transect Surveyed: 03/19/02

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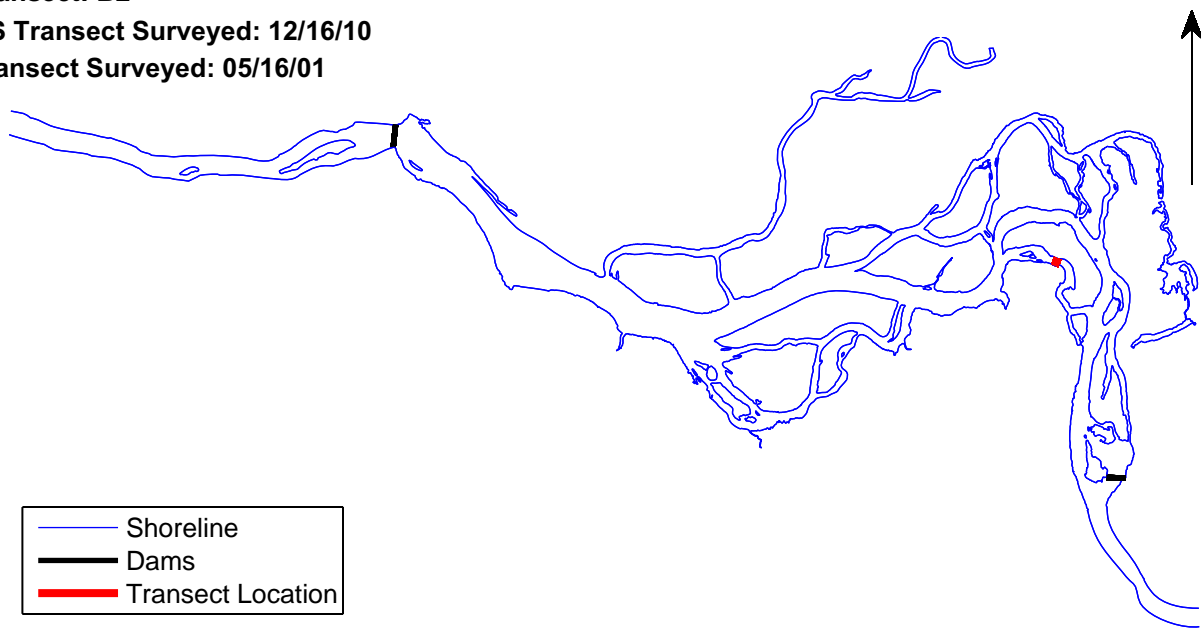


Orientation Looking Downstream

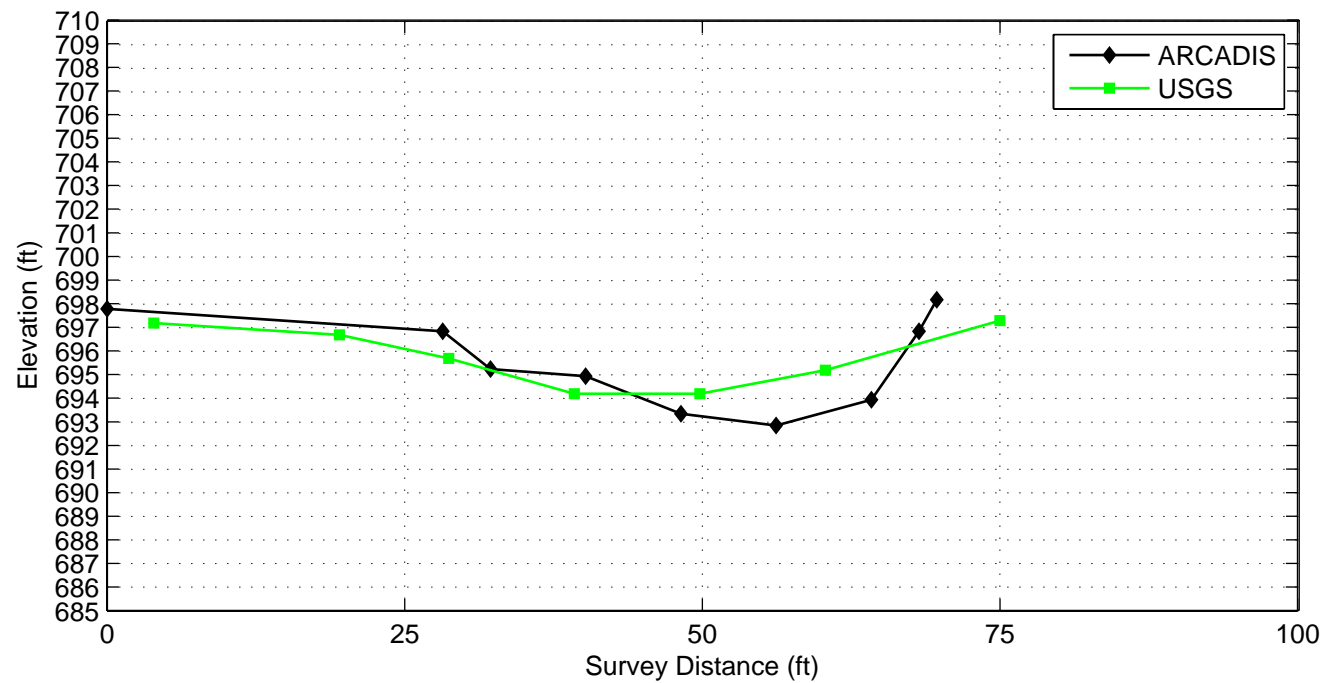


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-38
USGS Transect: B2
ARCADIS Transect Surveyed: 12/16/10
USGS Transect Surveyed: 05/16/01

DRAFT

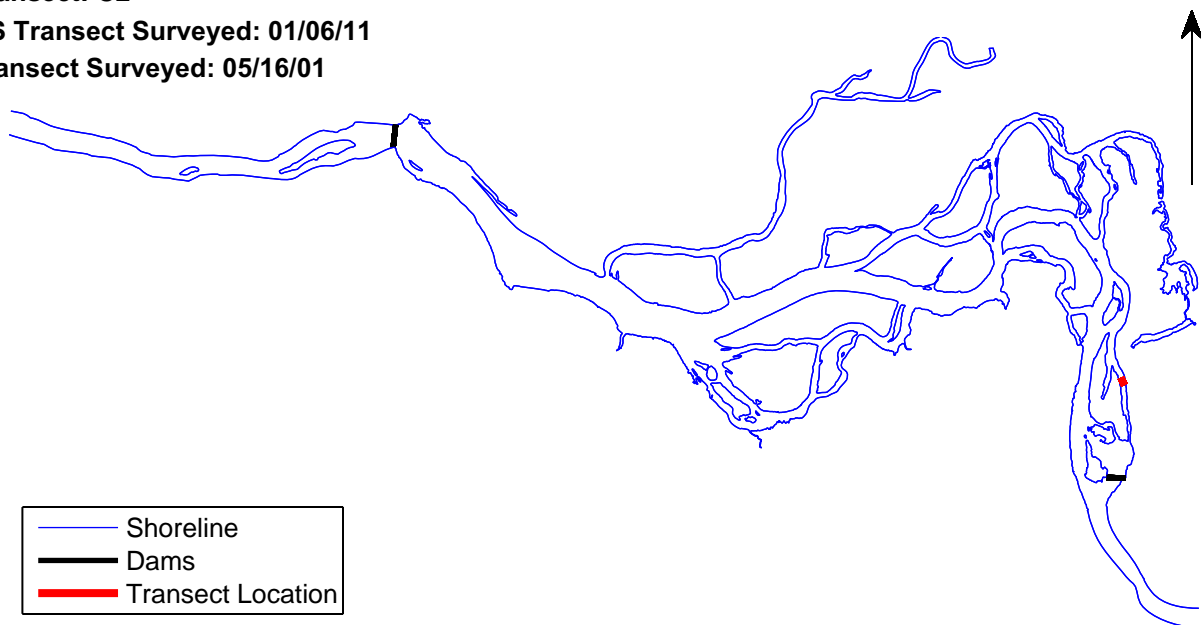


Orientation Looking Downstream

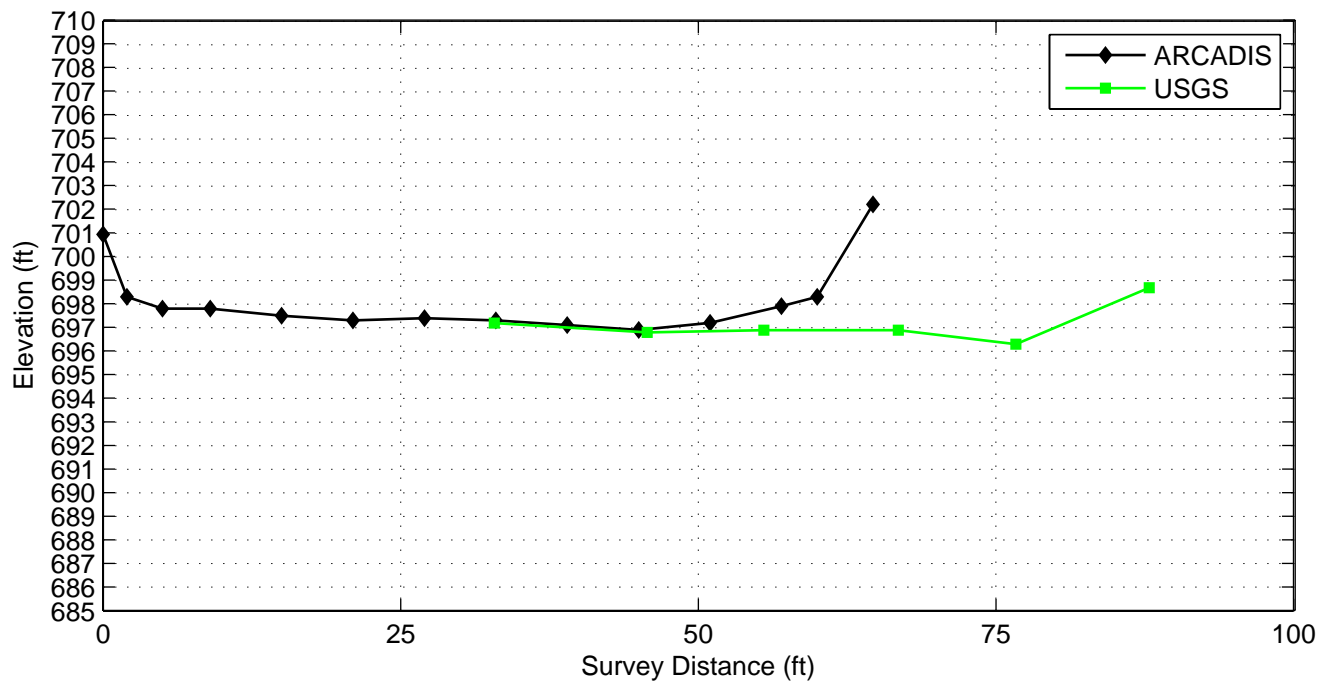


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-40
USGS Transect: C2
ARCADIS Transect Surveyed: 01/06/11
USGS Transect Surveyed: 05/16/01

DRAFT

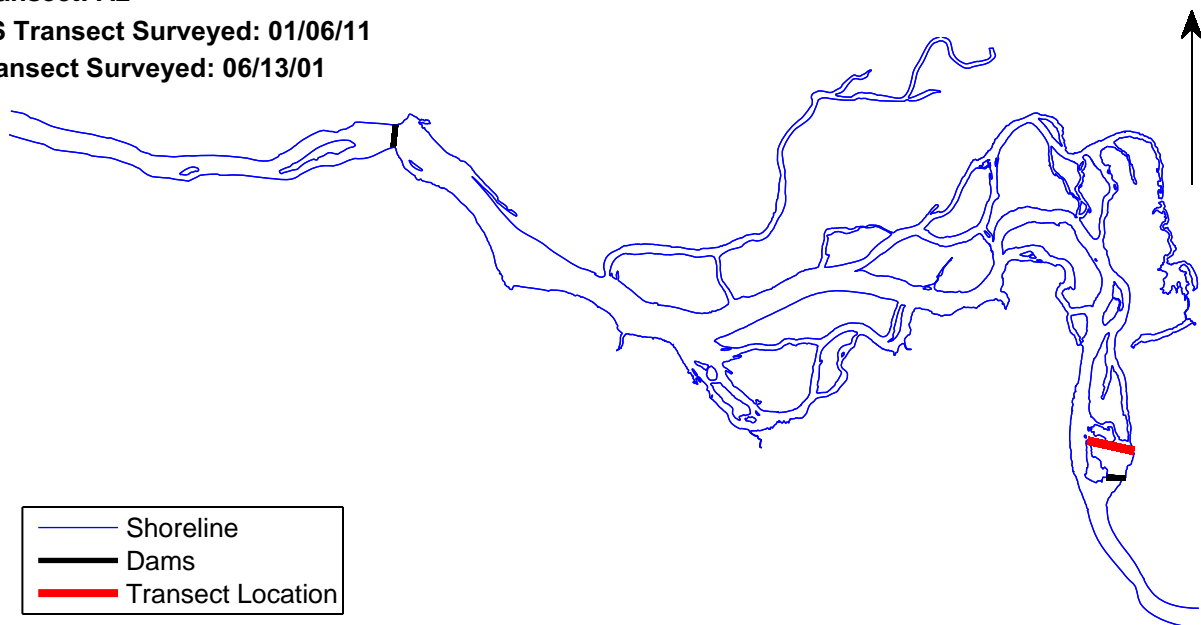


Orientation Looking Downstream

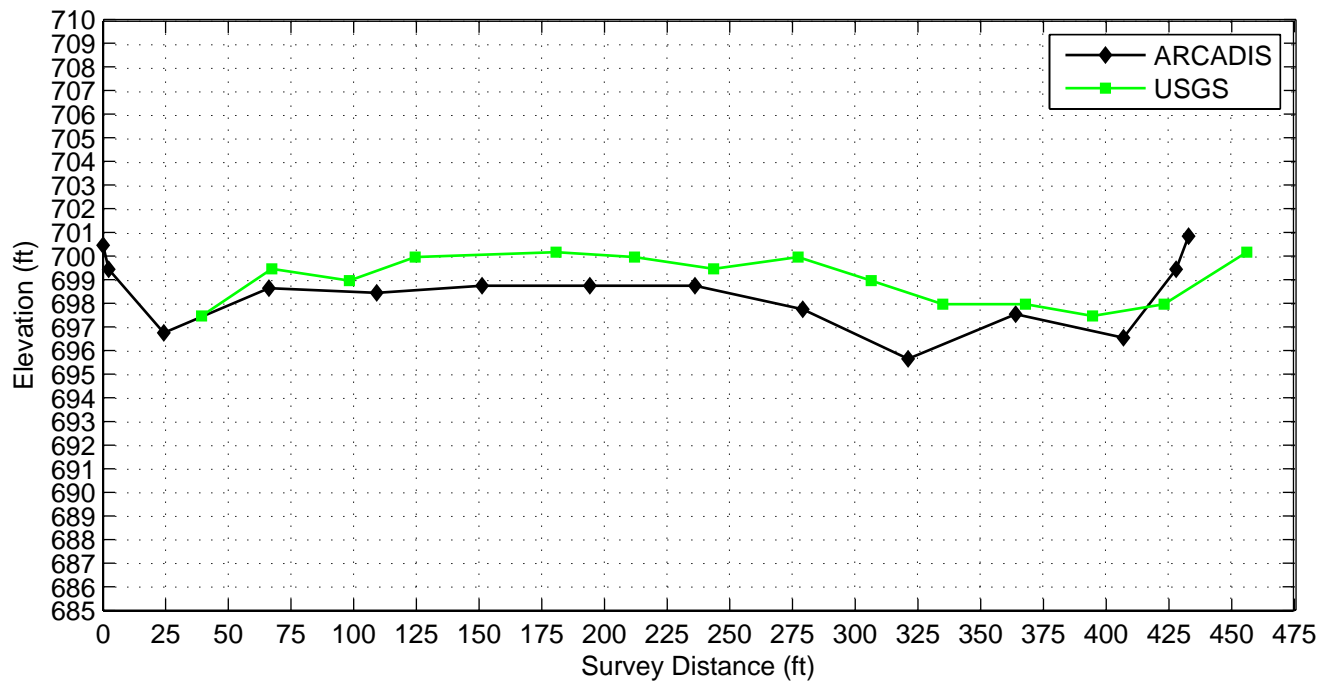


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-42
USGS Transect: A2
ARCADIS Transect Surveyed: 01/06/11
USGS Transect Surveyed: 06/13/01

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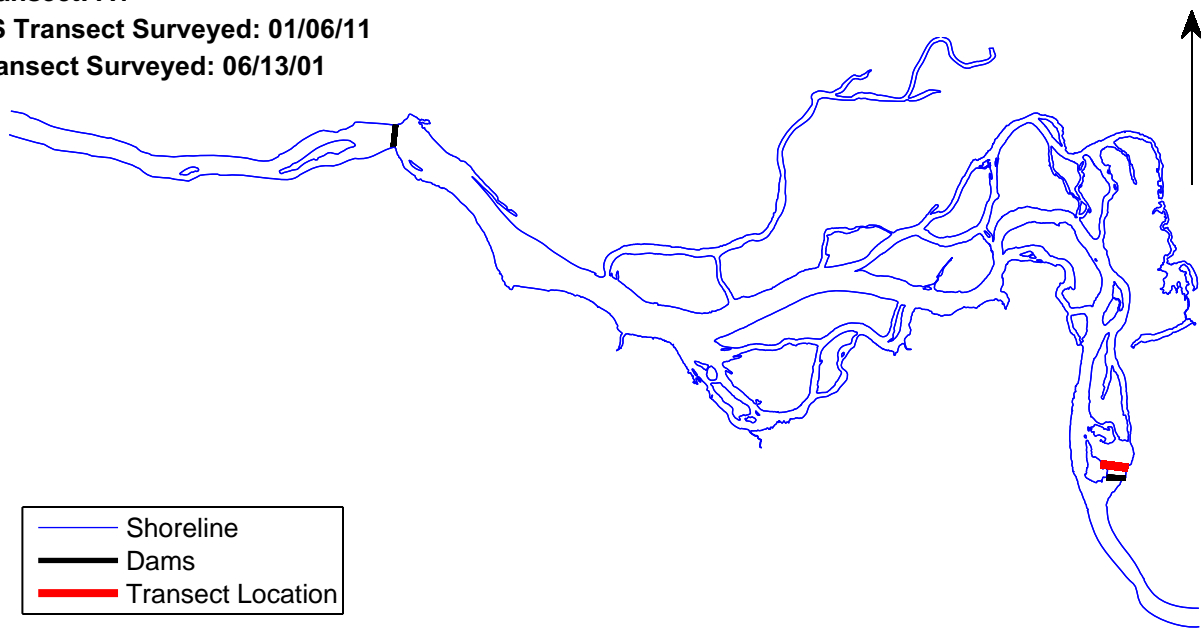


Orientation Looking Downstream

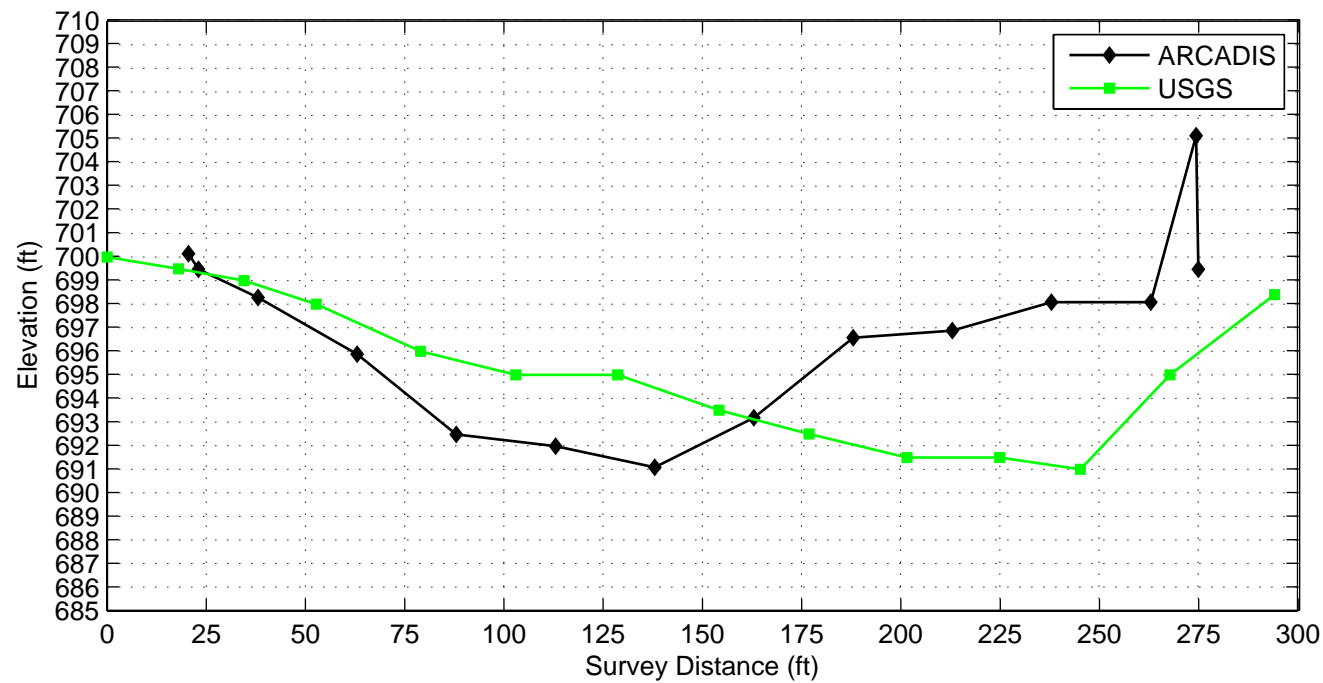


Otsego City Area 2 Bank Profiles
ARCADIS Transect: OCRT-43
USGS Transect: A1
ARCADIS Transect Surveyed: 01/06/11
USGS Transect Surveyed: 06/13/01

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Orientation Looking Downstream



Attachment C

Area 2 Non-PCB Sampling
Information

Georgia-Pacific LLC
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Supplemental Remedial Investigations/Feasibility Studies
Area 2 Soil and Sediment Sampling Plan

Table C-1 -- Floodplain Soil TCL/TAL Data

Sampling Event	1993 Floodplain Investigation	1993 Floodplain Investigation
Location ID	KF3-1	KF3-1
Northing	351846.91	351846.91
Easting	12768391.43	12768391.43
Elevation	697.67	697.67
Rivermile	53.53	53.53
SRI Reach	AREA 2	AREA 2
RI Reach	A2	A2
Sample ID	K10053	K10054
Sample Date	7/9/1993	7/9/1993
Depth Interval (in)	0-6	6-12
Inorganics (mg/kg)		
Aluminum	12,000	8,800
Antimony	41 UJ [12 UJ]	54 UJ
Arsenic	13 J	14 BJ
Barium	220	220
Beryllium	0.76 B	0.97 U
Cadmium	3.8 BJ	6.5 J
Calcium	28,000	18,000
Chromium	150	100
Cobalt	11 B	6.8 B
Copper	160 J	130 J
Cyanide	0.32 U	0.58 U
Iron	26,000	19,000
Lead	360	460
Magnesium	8,200	3,000 B
Manganese	460	190
Mercury	1.3	2.0
Nickel	52	36 B
Potassium	620 U	810 U
Selenium	2.3 BJ	2.2 UJ
Silver	-	5.9 U
Sodium	780 U	1,000 U
Thallium	2.1 U	3.8 U
Vanadium	25 B	22 B
Zinc	460	330

Pesticides (mg/kg)		
4,4'-DDD	0.014 U	0.025 U
4,4'-DDE	0.014 J	0.025 U
4,4'-DDT	0.014 U	0.025 U
Aldrin	0.022	0.011 J
Alpha-BHC	0.0073 U	0.013 U
Beta-BHC	0.0098 JN	0.013 U
Delta-BHC	0.0073 U	0.013 U
Gamma-BHC (Lindane)	0.0073 U	0.013 U
Alpha-Chlordane	0.014 U	0.025 U
Gamma-Chlordane	0.0062 JN	0.013 U
Dieldrin	0.014 U	0.025 U
Endosulfan I	0.014 U	0.025 U
Endosulfan II	0.014 U	0.025 U
Endosulfan Sulfate	0.013 JN	0.025 U
Endrin	0.014 U	0.025 U
Endrin Aldehyde	0.0073 U	0.013 U
Endrin Ketone	0.0082 J	0.013 U
Heptachlor	0.0073 U	0.013 U
Heptachlor Epoxide	0.0073 U	0.013 U
Methoxychlor	0.073 U	0.13 U
Toxaphene	0.73 U	1.3 U

Semivolatiles (mg/kg)		
2,2'-Oxybis(1-Chloropropane)	1.4 U	2.5 U
2,4,5-Trichlorophenol	3.4 U	6.1 U
2,4,6-Trichlorophenol	1.4 U	2.5 U
2,4-Dichlorophenol	1.4 U	2.5 U
2,4-Dimethylphenol	1.4 U	2.5 U
2,4-Dinitrophenol	3.4 UJ	6.1 UJ
2,4-Dinitrotoluene	1.4 U	2.5 U
2,6-Dinitrotoluene	1.4 U	2.5 U
2-Chloronaphthalene	1.4 U	2.5 U
2-Chlorophenol	1.4 U	2.5 U
2-Methylnaphthalene	1.4 U	2.5 U
2-Methylphenol	1.4 U	2.5 U
2-Nitroaniline	3.4 U	6.1 U
2-Nitrophenol	1.4 U	2.5 U
3,3'-Dichlorobenzidine	1.4 U	2.5 U
3-Nitroaniline	3.4 U	6.1 U
4,6-Dinitro-2-methylphenol	3.4 U	6.1 U
4-Bromophenyl-phenylether	1.4 U	2.5 U
4-Chloro-3-Methylphenol	1.4 U	2.5 U
4-Chloroaniline	1.4 UJ	2.5 UJ
4-Chlorophenyl-phenylether	1.4 U	2.5 U
4-Methylphenol	1.4 U	2.5 U
4-Nitroaniline	3.4 U	6.1 U
4-Nitrophenol	3.4 U	6.1 U
Acenaphthene	1.4 U	2.5 U
Acenaphthylene	1.4 U	2.5 U
Anthracene	1.4 U	2.5 U
Benzo(a)anthracene	0.34 J	2.5 U
Benzo(a)pyrene	1.4 U	2.5 U
Benzo(b)fluoranthene	1.4 U	2.5 U
Benzo(g,h,i)perylene	1.4 U	2.5 U
Benzo(k)fluoranthene	1.4 U	2.5 U
bis(2-Chloroethoxy)methane	1.4 U	2.5 U
bis(2-Chloroethyl)ether	1.4 U	2.5 U
bis(2-Ethylhexyl)phthalate	0.32 J	2.5 U
Butylbenzylphthalate	1.4 U	2.5 U
Carbazole	1.4 U	2.5 U
Chrysene	0.42 J	2.5 U
Dibenzo(a,h)anthracene	1.4 U	2.5 U
Dibenzofuran	1.4 U	2.5 U
Diethylphthalate	1.4 U	2.5 U
Dimethylphthalate	1.4 U	2.5 U
Di-n-Butylphthalate	0.20 J	2.5 U
Di-n-Octylphthalate	1.4 U	2.5 U
Fluoranthene	0.62 J	0.27 J
Fluorene	1.4 U	2.5 U
Hexachlorobenzene	1.4 U	2.5 U
Hexachlorobutadiene	1.4 U	2.5 U
Hexachlorocyclopentadiene	1.4 U	2.5 U
Hexachloroethane	1.4 U	2.5 U
Indeno(1,2,3-cd)pyrene	1.4 U	2.5 U
Isophorone	1.4 U	2.5 U
Naphthalene	1.4 U	2.5 U
Nitrobenzene	1.4 U	2.5 U
N-Nitroso-di-n-propylamine	1.4 U	2.5 U
N-Nitrosodiphenylamine	1.4 U	2.5 U
Pentachlorophenol	3.4 U	6.1 U
Phenanthrene	0.30 J	2.5 U
Phenol	1.4 U	2.5 U
Pyrene	0.55 J	0.25 J

Volatiles (mg/kg)		
1,1,1-Trichloroethane	0.045 U	0.071 U
1,1,2,2-Tetrachloroethane	0.045 U	0.071 U
1,1,2-Trichloroethane	0.045 U	0.071 U
1,1-Dichloroethane	0.045 U	0.071 U
1,1-Dichloroethene	0.045 U	0.071 U
1,2,4-Trichlorobenzene	1.4 U	2.5 U
1,2-Dichlorobenzene	1.4 U	2.5 U
1,2-Dichloroethane	0.045 U	0.071 U
1,2-Dichloroethene (total)	0.045 U	0.071 U
1,2-Dichloropropane	0.045 U	0.071 U
1,3-Dichlorobenzene	1.4 U	2.5 U
1,4-Dichlorobenzene	1.4 U	2.5 U
2-Butanone	0.045 U	0.071 U
2-Hexanone	0.045 U	0.071 U
4-Methyl-2-pentanone	0.045 U	0.071 U
Acetone	0.049	0.071 UJ
Benzene	0.045 U	0.071 U
Bromodichloromethane	0.045 U	0.071 U
Bromoform	0.045 U	0.071 U
Bromomethane	0.045 U	0.071 U
Carbon Disulfide	0.045 U	0.071 U
Carbon Tetrachloride	0.045 U	0.071 U
Chlorobenzene	0.045 U	0.071 U
Chloroethane	0.045 U	0.071 U
Chloroform	0.045 U	0.071 U
Chloromethane	0.045 U	0.071 U
cis-1,3-Dichloropropene	0.045 U	0.071 U
Dibromochloromethane	0.045 U	0.071 U
Ethylbenzene	0.045 U	0.071 U
Methylene Chloride	0.045 U	0.071 U
Styrene	0.045 U	0.071 U
Tetrachloroethene	0.045 U	0.071 U
Toluene	0.045 U	0.071 U
trans-1,3-Dichloropropene	0.045 U	0.071 U
Trichloroethene	0.045 U	0.071 U
Vinyl Chloride	0.045 U	0.071 U
Xylenes (total)	0.045 U	0.071 U

Notes:

■ - Grey shading indicates the presence of a non-detect result.

mg/kg - milligrams per kilogram

U The compound was analyzed for but not detected. The associated value is the compound quantitation limit.

B The compound was found in the sample as well as its associated blank, its presence in the sample may be suspect.

J The compound was positively identified; however, the associated numerical value is an estimated concentration only.

UJ The compound was not detected above the reported sample quantitation limit. However, the reported limit is approximate and may or may not represent the actual limit of quantitation.

JN The analysis indicates the presence of a compound for which there is presumptive evidence to make a tentative identification. The associated numerical value is an estimated concentration only.

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Table C-2 -- USEPA Floodplain Soil Dioxin/Furans Data

Compound	SL055 (Soil)	SL058 (Soil)	SL060 (Soil)	SL065 (Soil)
	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹
2,3,7,8-TCDD	ND	ND	16	ND
1,2,3,7,8-PeCDD	ND	ND	ND	ND
1,2,3,4,7,8-HxCDD	1.7	24	7.8	ND
1,2,3,6,7,8-HxCDD	14	110	39	ND
1,2,3,7,8,9-HxCDD	3.4	56	19	ND
1,2,3,4,6,7,8-HpCDD	920	2,800	1,300	30
OCDD	10,000	24,000	9,300	240
2,3,7,8-TCDF	19	ND	49	ND
1,2,3,7,8-PeCDF	0.76	12	8.7	ND
2,3,4,7,8-PeCDF	1.1	ND	7.2	ND
1,2,3,4,7,8-HxCDF	1.3	27	14	ND
1,2,3,6,7,8-HxCDF	6.1	55	27	ND
1,2,3,7,8,9-HxCDF	ND	ND	ND	ND
2,3,4,6,7,8-HxCDF	0.43	7.1	ND	ND
1,2,3,4,6,7,8-HpCDF	40	480	230	14
1,2,3,4,7,8,9-HpCDF	2.6	20	9.8	ND
OCDF	160	490	280	48

Notes:

1. For presentation purposes, results rounded to 2-significant figures.

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Table C-3 -- USEPA Sediment Dioxin/Furans Data

Compound	SD026 (Sediment)	SD027 (Sediment)	SD029 (Sediment)	SD035 (Sediment)	SD040 (Sediment)	SD049 (Sediment)	SD051 (Sediment)	SD052 (Sediment)	SD055 (Sediment)
	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹	Sample Concentration (ng/kg) ¹
2,3,7,8-TCDD	ND	ND	ND	ND	ND	ND	ND	33	ND
1,2,3,7,8-PeCDD	ND	ND	15	ND	ND	ND	ND	ND	ND
1,2,3,4,7,8-HxCDD	ND	49	52	ND	ND	ND	ND	27	85
1,2,3,6,7,8-HxCDD	ND	230	270	36	ND	14	190	230	700
1,2,3,7,8,9-HxCDD	ND	83	100	ND	ND	ND	73	110	310
1,2,3,4,6,7,8-HpCDD	14	9,200	8,100	1,000	ND	440	7,300	7,700	22,000
OCDD	120	86,000	88,000	8,800	26	3,200	60,000	52,000	150,000
2,3,7,8-TCDF	21	82	440	ND	ND	ND	220	140	340
1,2,3,7,8-PeCDF	ND	ND	38	ND	ND	ND	ND	ND	190
2,3,4,7,8-PeCDF	ND	16	29	37	ND	ND	25	21	150
1,2,3,4,7,8-HxCDF	6.1	66	140	ND	ND	ND	49	52	350
1,2,3,6,7,8-HxCDF	ND	120	150	24	ND	9.7	140	180	140
1,2,3,7,8,9-HxCDF	ND	ND	20	ND	ND	ND	ND	16	ND
2,3,4,6,7,8-HxCDF	ND	16	23	ND	ND	ND	ND	22	50
1,2,3,4,6,7,8-HpCDF	ND	1,600	1,000	230	ND	100	1,700	2,200	7,300
1,2,3,4,7,8,9-HpCDF	ND	70	77	490	ND	ND	65	39	170
OCDF	330	4,000	2,000	230	ND	130	2,600	1,800	8,200

Notes:

1. For presentation purposes, results rounded to 2-significant figures.

TestAmerica Standard
Operating Procedure for
Analysis of Dioxin/Furans

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TESTAMERICA KNOXVILLE

STANDARD OPERATING PROCEDURE

TITLE: Analysis of Polychlorinated Dioxins/Furans by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) Based on Methods 8290, 8290A, 1613B, 23, 0023A, and TO-9A

(SUPERSEDES: KNOX-ID-0004, Rev. 7)

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1. Scope and Application

- 1.1 This procedure is used for the determination of tetra- through octa- chlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) in water, soils, solids, sediments, wipes, biological samples, fly ash, XAD resin, filters, still bottoms, waste oils, and other sample matrices by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). This procedure is designed to meet analytical program requirements where US EPA Method 8290, 8290A, 1613B, 23, 0023A, or TO-9A is specified.
- 1.2 The seventeen 2,3,7,8-substituted and total Tetra-Hepta PCDDs/PCDFs listed in Table 1 can be determined by this procedure. Specifications are also provided for separate determination of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) and 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF). In addition, total homologs (i.e., Total TCDD, Total TCDF, etc.) can be identified by this method.
- 1.3 The detection limits and quantitation levels in this method are usually dependent on the level of interferences rather than instrumental limitations. The minimum levels (MLs) in Table 2 are the levels at which the PCDDs/PCDFs can be quantitated with no interferences present.
- 1.4 This procedure is designed for use by analysts who are experienced with residue analysis and skilled in HRGC/HRMS. Each analyst must demonstrate the ability to generate acceptable results with this method.
- 1.5 Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCDDs or PCDFs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed. Section 5 of this procedure discusses safety procedures.

2. Summary of Method

- 2.1 This procedure uses high resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques.
- 2.2 Samples are spiked with a solution of known amounts of the isotopically labeled internal standards listed in Table 13 and Table 15. The samples are then extracted using matrix specific extraction procedures.
 - 2.2.1 Water samples are extracted using separatory funnel techniques with methylene chloride as the extraction solvent.
 - 2.2.2 Solid samples are extracted by Soxhlet extraction with the appropriate solvent.
 - 2.2.3 Organic liquid waste samples are diluted in solvent.
- 2.3 After extraction, the sample is concentrated and solvent exchanged with hexane. The extract is then subjected to one or more cleanup steps to remove the sample of interferences. The final extract is prepared by adding a known amount of the labeled recovery standards (13C12-1,2,3,4-TCDD and 13C12-1,2,3,7,8,9-HxCDD) and concentrating to the final volume.
- 2.4 The acid-base cleanup of the sample is used before column chromatography for samples that contain large amounts of basic and acid coextractables. If such interferences are not removed

before column chromatography, they can cause a shift in the predicted elution pattern. Conditions which can indicate the need for this procedure are: samples which are highly colored, samples which contain lipids or other oxidizable compounds or samples which contain known large amounts of polar organics.

- 2.5 Silica gel is effective in removing chlorophenoxy herbicide residues, while alumina partitions PCBs, 2,4,5-trichlorophenol and hexachlorophene.
- 2.6 When the above cleanup techniques do not completely remove interferences, an activated carbon cleanup is used to remove interferences.
- 2.7 An aliquot of the extract is injected into the gas chromatograph. The analytes are separated by the GC and detected by a high resolution ($\geq 10,000$) mass spectrometer. Two exact m/z 's are monitored for each analyte.
- 2.8 The identification of the target 2,3,7,8 substituted isomers is based on their retention time relative to the labeled internal standards as established during routine calibration and the simultaneous detection of the two most abundant ions in the molecular ion region. All other PCDD/PCDF congeners are identified by their retention times falling within retention time windows as established during routine calibration, and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation of identification is based on comparing the calculated ion ratios with the theoretical ion abundances. The identification of 2,3,7,8-TCDF is confirmed on an isomer specific (DB-225) GC column.
- 2.9 Quantitation of the 2,3,7,8-substituted PCDD/PCDF isomers, total PCDDs, and total PCDFs is based on their relative response to the internal standards. A multipoint calibration is performed to establish mean response factors for the target analytes. The instrument performance is routinely checked by the analysis of continuing calibration standards. Method performance is demonstrated by the analysis of method blanks, initial precision and recovery samples, and ongoing precision and recovery samples.

3. Definitions

- 3.1 Analyte: A PCDD or PCDF tested for by this method. The analytes are listed in Table 1.
- 3.2 Calibration Standard: A solution prepared from a secondary standard and/or stock solution and used to calibrate the response of the instrument with respect to analyte concentration.
- 3.3 Calibration Verification Standard (VER): The mid-point calibration standard (CS3) that is used to verify calibration. See Table 5 and Table 6.
- 3.4 Cleanup Standard: $^{37}\text{Cl}_4$ -2,3,7,8-TCDD which is added to samples, blanks, quality control samples, and calibration solutions. It is added to the samples after extraction but prior to extract cleanup, and is used to judge the efficiency of the cleanup procedures.
- 3.5 Column Performance Solution Mixture (CPSM): A mixture of TCDD or TCDF isomers (including the 2,3,7,8-TCDD or 2,3,7,8-TCDF isomer) known to elute close to the retention time of 2,3,7,8-TCDD or 2,3,7,8-TCDF on the analytical column being used. It is used to demonstrate acceptable resolution between the 2,3,7,8-TCDD or 2,3,7,8-TCDF isomer and all other TCDD or TCDF isomers on analytical column (percent valley $< 25\%$).

- 3.6 Congener: Any member of a particular homologous series, for example, 1,2,3,7,8-pentachlorodibenzofuran.
- 3.7 CS1, CS2, CS3, CS4, CS5: See Calibration Standard and Table 5 and Table 6.
- 3.8 Detection Limit (DL): The minimum concentration of the target analyte that can be detected. Sample specific detection limits are calculated from the instrument noise level and internal standard response.
- 3.9 Estimated Detection Limit (EDL): The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level.
- 3.10 Estimated Maximum Possible Concentration (EMPC): The calculated concentration of a signal in the same retention time region as a target analyte but which does not meet the other qualitative identification criteria defined in the procedure.
- 3.11 GC: Gas chromatograph or gas chromatography
- 3.12 Homologous Series: A series of compounds in which each member differs from the next member by a constant amount. The members of the series are called homologs.
- 3.13 HRGC: High resolution GC
- 3.14 HRMS: High resolution MS
- 3.15 ICV: Initial Calibration Verification Standard. A calibration standard from a second source, traceable to a national standard if possible. The ICV is analyzed after the initial calibration to verify the concentration of the Initial Calibration Standards.
- 3.16 Internal Standards: Isotopically labeled analogs of the target analytes that are added to every sample, blank, quality control spike sample, and calibration solution. They are added to the sample before extraction and are used to calculate the concentration of the target analytes or detection limits.
- 3.17 IPR: Initial precision and recovery; four aliquots of the diluted PAR standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed prior to the first time this method is used and any time the method or instrumentation is modified.
- 3.18 Isomer: Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are structural isomers.
- 3.19 Laboratory Blank: See Method Blank.
- 3.20 Laboratory Control Sample: See Ongoing Precision and Recovery Standard (OPR).
- 3.21 Maximum Level (MaxL): The concentration or mass of analyte in the sample that corresponds to the highest calibration level in the initial calibration. Also referred to as the upper method calibration limit (UMCL). It is equivalent to the concentration of the highest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

- 3.22 Method Blank: An aliquot of reagent water, sand, sodium sulfate, or other representative matrix, free of the targets of interest and interferences, that is extracted and analyzed along with the samples to monitor for laboratory contamination.
- 3.23 Minimum Level (MinL): The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. Also referred to as the lower method calibration limit (LMCL). It is equivalent to the concentration of the lowest calibration standard assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.
- 3.24 MS: Mass spectrometer or mass spectrometry.
- 3.25 Multiple Ion Detection (MID): A MS operational mode in which only selected ions are monitored rather than scanning the instrument to obtain a complete mass spectrum.
- 3.26 OPR: Ongoing precision and recovery standard; a laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.
- 3.27 PAR: Precision and recovery standard; secondary standard that is diluted and spiked to form the IPR and OPR.
- 3.28 PCDD: Polychlorinated dibenzo-p-dioxins.
- 3.29 PCDF: Polychlorinated dibenzofurans.
- 3.30 PFK: Perfluorokerosene; the mixture of compounds used to calibrate the exact m/z scale in the HRMS.
- 3.31 Recovery Standard: $^{13}\text{C}_{12}$ -1,2,3,4-TCDD and $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD which are added to every sample, blank, and quality control spike sample extract prior to analysis. They are used to measure the recovery of the internal standards and the cleanup standard.
- 3.32 Relative Percent Difference (RPD): A measure of the difference between two values normalized to one of the values. It is used to determine the accuracy of the concentration measurements of second source verification standards.
- 3.33 Relative Response Factor (RRF): The ratio of the response of the mass spectrometer to a known amount of a compound relative to that of a known amount of a reference standard as measured in the initial and continuing calibrations. It is used to determine instrument performance and it is used to calculate the concentration of target analytes, internal standard recoveries, or detection limits in samples, blanks, and quality control samples.
- 3.34 Signal to Noise Ratio: The ratio of the mass spectrometer response of a GC peak to the background noise signal.
- 3.35 Split Ratio (S): The decimal expression of the proportion of extract used from splits taken after the addition of internal standards and before the addition of recovery standards.

3.36 Window Defining Mix: A solution which contains the first and last eluting isomers of each homologue group and is used to verify that the switching times between the MID descriptors have been appropriately set.

3.37 Additional definitions can be found in the Test America Knoxville QAM

4. Interferences

- 4.1 Solvents, reagents, glassware and other sample processing hardware can yield discrete artifacts or elevated baselines that can cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferences under the conditions of analysis by performing laboratory method blanks. Analysts must avoid using PVC gloves, powdered gloves, or gloves with levels of phthalates which cause interference.
- 4.2 The use of high purity reagents and solvents (pesticide grade) helps minimize interference problems. Where necessary, reagents are cleaned by extraction or solvent rinse.
- 4.3 Interferences coextracted from the samples can vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated alkyldibenzofurans that can be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. While certain cleanup techniques are provided as part of this method, unique samples can require additional cleanup steps to achieve lower detection limits.

5. Safety

- 5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.
- 5.2 Eye protection that satisfies ANSI Z87.1 (as per the Test America Health and Safety Manual), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated must be removed and discarded, other gloves must be cleaned immediately.
 - 5.2.1 Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. For the operations described herein, Nitrile clean room gloves are worn. For operations using solvents that splash, silver shield gloves are recommended. Silver shield gloves protect against breakthrough for most of the solvents used in this procedure
- 5.3 Finely divided dry soils contaminated with PCDDs and PCDFs can be particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.
- 5.4 The effluents of sample splitters for the gas chromatograph and roughing pumps on the mass spectrometer must be vented to the laboratory hood exhaust system or must pass through an activated charcoal filter.

- 5.5 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them or use thermal protection when working on them while they are above room temperature.
- 5.6 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. Alternatively, the source can be removed from the vacuum manifold through a vacuum interlock.
- 5.7 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power. If the work involved requires measurement of voltage supplies, the instrument can be left on.
- 5.8 When using a scalpel, cut away from yourself. If you are holding something, cut away from your hand.
- 5.9 Equipment goggles or a face shield must be used when employees are using solvents to rinse or clean glassware.
- 5.10 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid (1)	Corrosive, Oxidizer, Dehydrator	1 mg/m ³	This material will cause burns if it comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive, Poison	2 ppm, 5 mg/m ³	This material will cause burns if it comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Hydrochloric Acid	Corrosive, Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and can cause damage to the eyes. Contact can cause severe burns and permanent eye damage.
Methylene chloride	Carcinogen, Irritant	25 ppm-TWA, 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. Can be absorbed through skin.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable, Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure can cause lightheadedness, nausea, headache, and blurred vision. Vapors can cause irritation to the skin and eyes.
Methanol	Flammable, Poison, Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure can include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and can cause skin to become dry and cracked. Skin absorption can occur; symptoms can parallel inhalation exposure. Irritant to the eyes.
Toluene	Flammable, Poison, Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation can cause irritation of the upper respiratory tract. Symptoms of overexposure can include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness can be produced. Causes severe eye and skin irritation with redness and pain. Can be absorbed through the skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Can cause coughing, dizziness, dullness, and headache.
Cyclohexane	Flammable, Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms can include coughing, shortness of breath. High concentrations have a narcotic effect.
Tetradecane	Irritant	None established	Inhalation of vapors can cause difficulty breathing, headache, intoxication and central nervous system damage.
Benzene	Flammable, Toxic, Carcinogen	PEL: 1 ppm TWA ; 5 ppm, 15 min. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations can have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim can experience tightness in the chest, breathlessness, and loss of consciousness.
Nonane	Flammable	None established	Harmful if inhaled/swallowed. Vapor/mist is irritating to eyes, mucous membranes and upper respiratory tract. Causes skin irritation.
Potassium Hydroxide	Corrosive, Poison	2 mg/m3 ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on the severity of exposure. Symptoms can include coughing, sneezing, and damage to the nasal or respiratory tract. High concentrations can cause lung damage. Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures.
1 – Always add acid to water to prevent violent reactions.			

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.10.1 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include benzene and methylene chloride, 2,3,7,8-TCDD and all other 2,3,7,8-substituted PCDD or PCDF isomers.

Note: The 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities comparable to that of 2,3,7,8-TCDD. The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.

- 5.11 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers must be kept closed unless transfers are being made.
- 5.12 The preparation of all standards and reagents and glassware cleaning procedures that involve solvents such as acetone, toluene, methylene chloride, and hexane must be conducted in a fume hood with the sash closed as far as the operations permits.
- 5.13 All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported immediately to a laboratory supervisor.
- 5.14 Training: Workers must complete the employee Corporate Safety Manual safety orientation prior to working in the laboratory.
- 5.15 Personal Hygiene: Thorough washing of hands and forearms is recommended after each manipulation and before breaks (coffee, lunch, and shifts).
- 5.16 Confinement: Work areas must be isolated and posted with signs. Glassware and tools must be segregated. Benchtops must be covered with plastic backed absorbent paper.
- 5.17 Waste: Good technique includes minimizing contaminated waste.
- 5.18 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

6. Equipment and Supplies

6.1 Sample Extraction Equipment.

Note: All glassware used in extraction and cleanup procedures is solvent rinsed twice before use with acetone, toluene, methylene chloride and hexane in that order. Pre-extract the Soxhlet apparatus with toluene for at least 4 hours. Rinse glassware with all 4 solvents once. See SOP KNOX-QA-0002, current revision, "Glassware Cleaning", for details.

6.1.1 Aqueous Sample Extraction

- 6.1.1.1 Multi-position separatory funnel rotator.
- 6.1.1.2 2000 mL separatory funnels with PTFE stopcocks and PTFE stoppers.
- 6.1.1.3 100 mm glass funnels with short stems.
- 6.1.1.4 Class A 1 mL pipettes.
- 6.1.1.5 Class A 1000 mL graduated cylinders.
- 6.1.1.6 PTFE squirt bottles, 500 mL.
- 6.1.1.7 Syringes.
- 6.1.1.8 Glass wool, precleaned with methylene chloride.
- 6.1.1.9 Buchner funnels, filter flasks, rubber stopper and GF/D filters
- 6.1.1.10 Vacuum source.

6.1.2 Soxhlet Extraction

- 6.1.2.1 Analytical balance, capable of weighing to 0.01 g.
- 6.1.2.2 Stainless steel spatula.
- 6.1.2.3 Stainless steel tweezers.
- 6.1.2.4 Soxhlet extractor or Dean-Stark Soxhlet extractor.
- 6.1.2.5 Heating mantles with temperature controls.
- 6.1.2.6 500 mL evaporative flask, round bottom.
- 6.1.2.7 Glass condenser, compatible with the dean-stark extractor.
- 6.1.2.8 Class A 1 mL pipettes.
- 6.1.2.9 Glass wool, precleaned with methylene chloride.
- 6.1.2.10 High purity glass fiber Soxhlet thimble.
- 6.1.2.11 Boiling beads, 6 mm glass.
- 6.1.2.12 PTFE boiling chips.

6.1.3 Waste Dilution

- 6.1.3.1 Analytical balance, capable of weighing to 0.01 g.
- 6.1.3.2 40 mL vial, with PTFE lined cap.
- 6.1.3.3 5 ¾ inch borosilicate glass pipets.

6.1.3.4 Rubber bulbs.

6.1.3.5 1 ml Class A pipette.

6.2 Sample Cleanup Equipment.

6.2.1 Acid-base cleanup

6.2.1.1 Disposable Pasteur pipets and rubber bulbs.

6.2.1.2 Graduated cylinder, 100 mL volume.

6.2.1.3 Vials, 40 mL volume, with PFTE lined caps.

6.2.2 Dual column cleanup

6.2.2.1 Disposable glass columns.

6.2.2.1.1 20mm x 240mm custom glass column with support ring and tapered tip.

6.2.2.1.2 16mm x 240mm custom glass column with support ring and tapered tip.

6.2.2.2 Aluminum support rack for custom columns.

6.2.2.3 Amber-colored glass jar with a PFTE lined screw cap, 250mL.

6.2.2.4 Disposable Pasteur pipets and rubber bulbs.

6.2.2.5 Bottletop solvent dispenser.

6.2.2.6 40 mL vials with PFTE lined screw caps.

6.2.2.7 Graduated cylinder, 100 ml.

6.2.2.8 Solvent waste collection jars, 125mL.

6.2.3 Activated carbon cleanup

6.2.3.1 10 mL disposable pipet for use as the column.

6.2.3.1.1 All disposable carbon columns are solvent rinsed before use. The solvents used are acetone, toluene, methylene chloride and hexane (in this order).

6.2.3.2 Glass wool, precleaned with methylene chloride.

6.2.3.3 25 mL graduated cylinder

6.2.3.4 40 mL vials.

6.3 Sample Concentration Equipment.

6.3.1 Macro Concentration Equipment – Rapid-Vap

- 6.3.1.1 Labconco Rapid-Vap concentrator
- 6.3.1.2 600 mL sample concentrator tubes, Labconco or equivalent.
- 6.3.1.3 Borosilicate 5.75 inch and 9.0 inch disposable pipettes.
- 6.3.1.4 Rubber bulbs.
- 6.3.1.5 Borosilicate 40 mL disposable vials with PFTE lined screwcaps.
- 6.3.2 Macro Concentration – Snyder Column
 - 6.3.2.1 Heating mantles with temperature controls.
 - 6.3.2.2 Three-ball macro Snyder column.
 - 6.3.2.3 Rubber bulbs.
 - 6.3.2.4 Nine inch borosilicate glass pipets.
 - 6.3.2.5 40 mL vial, with PFTE lined cap.
 - 6.3.2.6 PFTE boiling chips.
- 6.3.3 Micro Concentration – N-Evap
 - 6.3.3.1 Nitrogen blowdown apparatus (N-EVAP or equivalent).
 - 6.3.3.2 Hamilton syringe –25 uL.
 - 6.3.3.3 Mini vials, 1.1 mL capacity with a tapered bottom; with PFTE faced, rubber septa and screw caps.
- 6.4 Sample Analysis Equipment.
 - 6.4.1 Gas Chromatograph --- Must have splitless or on-column injection port for capillary column, temperature program with isothermal hold, and must meet all of the performance specification in Section 9.10.
 - 6.4.1.1 GC column for PCDDs/PCDFs and for isomer specificity for 2,3,7,8-TCDD --- 60m x 0.32mm ID x 0.25µm film thickness DB-5 or RTX-5 fused silica capillary column (J&W No. 123-5062, Restek No.10227 or 10227-125 IntegraGuard) or equivalent is required.
 - 6.4.1.2 GC column for isomer specificity for 2,3,7,8-TCDF --- 30m x 0.32mm ID x 0.25µm film thickness DB-225 or RTX-225 fused silica capillary column (J&W No. 123-2232 or Restek No.14024) or equivalent is required.
 - 6.4.2 Mass Spectrometer --- Electron impact ionization with the filament eV's optimized for best instrument sensitivity, stability and signal to noise ratio. Must be capable of repetitively selectively monitoring 12 exact m/z's minimum at high resolution (≥10,000) during a period of approximately 1 second and must meet all of the performance specifications in Section 9.10.

6.4.3 GC/MS Interface --- The mass spectrometer (MS) must be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beam

6.4.4 Data System --- Capable of collecting, recording, and storing MS data.

7. Reagents and Standards

7.1 Sample Pre-Treatment

7.1.1 Hydrochloric acid (HCl), concentrated 37% wt in water (ACS), Mallinckrodt AR Select or equivalent.

7.1.2 1N HCl - Carefully add 83mL of concentrated HCl to 917 mL of reagent water in a glass container.

7.2 Aqueous Extraction

7.2.1 Acetone, pesticide quality or equivalent.

7.2.2 Toluene, pesticide quality or equivalent.

7.2.3 Methylene chloride, pesticide quality or equivalent.

7.2.4 Hexane, pesticide quality or equivalent.

7.2.5 Tetradecane, pesticide quality or equivalent.

7.2.6 Reagent water must be produced by a Millipore DI system or equivalent, being able to produce water with 18 mega ohm (MΩ) resistance. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2.7 Sodium sulfate, reagent grade, granular, anhydrous (J.T Baker 3375, or equivalent)- Sodium Sulfate is cleaned by putting approximately 600g of Sodium Sulfate in large amber-colored glass jars and completely covering with methylene chloride, stirring the mixture with a stirring rod and letting the Sodium Sulfate soak for 5 minutes. The methylene chloride is drained and this step is repeated. After the methylene chloride is drained the Sodium Sulfate is transferred to a Buchner Funnel fitted onto a Vacuum Flask and is rinsed 2 times with methylene chloride while a vacuum is being applied to the apparatus. The Sodium Sulfate is then placed into shallow borosilicate glass dishes where it is allowed to dry. When the Sodium Sulfate is air dried it is placed in an oven at approximately 125 C – 135 C for one hour to drive off any residual moisture. After drying in the oven the Sodium Sulfate is transferred into pre-cleaned glass jars with fluoropolymer lined screw caps and is placed in a desiccator until needed.

7.3 Soxhlet extraction

7.3.1 Acetone, pesticide quality or equivalent.

7.3.2 Toluene, pesticide quality or equivalent.

7.3.3 Methylene chloride, pesticide quality or equivalent.

7.3.4 Hexane, pesticide quality or equivalent.

7.3.5 Benzene, pesticide quality or equivalent.

7.3.6 Tetradecane, pesticide quality or equivalent.

7.3.7 Sand, prepared by extracting with methylene chloride and/or baking at 450 °C for a minimum of 4 hours. After cooling store in a dessicator.

7.3.8 Sodium sulfate, reagent grade, granular, anhydrous (J.T Baker 3375, or equivalent)- Refer to Section 7.2.7 for details of sodium sulfate preparation.

7.3.9 Dry Ice, purchased from local vendor.

7.3.10 Reagent water must be produced by a Millipore DI system or equivalent, being able to produce water with 18 mega ohm (MΩ) resistance. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.4 Waste Dilution

7.4.1 Hexane, pesticide quality or equivalent.

7.4.2 Benzene, pesticide quality or equivalent.

7.5 Acid-Base Cleanup

7.5.1 Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.

7.5.2 Potassium hydroxide, 20% aqueous. Prepare by cautiously adding, 200 g of potassium hydroxide pellets to 800 mL of deionized water. This solution is stored at room temperature in a plastic bottle.

7.5.3 Sodium chloride, NaCl, analytical reagent, 5 percent (w/v) in reagent grade water.

7.5.4 Hexane, pesticide quality or equivalent.

7.5.5 Benzene, pesticide quality or equivalent.

7.5.6 Sodium sulfate, reagent grade, granular, anhydrous (J.T Baker 3375, or equivalent)- Refer to Section 7.2.7 for details of sodium sulfate preparation.

7.6 Silica Gel/Alumina Column Cleanup

7.6.1 Sodium sulfate, reagent grade, granular, anhydrous (J.T Baker 3375, or equivalent)- Refer to Section 7.2.7 for details of sodium sulfate preparation.

7.6.2 Methylene chloride pesticide quality or equivalent.

7.6.3 Hexane, pesticide quality or equivalent.

7.6.4 Acetone, pesticide quality or equivalent.

7.6.5 Toluene, pesticide quality or equivalent.

- 7.6.6 Silica gel, F679-212, Fisher Chromatographic Silica Gel, 100-200 mesh or equivalent. Prepare by Soxhlet extraction with methylene chloride for at least 6 hours. Transfer to a shallow, borosilicate glass dish and air dry. After drying, cover with aluminum foil and activate in an oven at 130°C for a minimum of four (4) hours. Store in labeled glass jars in a desiccator until use.
- 7.6.7 3.3% Deactivated silica gel – To prepare add 6.6 mL of reagent water to 200 g of silica gel (section 7.6.6) in a 250 mL amber-colored glass jar with a PFTE lined screw cap. Mix thoroughly by shaking until no lumps are visible, and the silica gel is free flowing and no longer sticks to the side of the jar.
- 7.6.8 Acidic silica gel - To prepare, add 57 mL of concentrated sulfuric acid to 180 g silica gel (section 7.6.6) in a 250 mL amber-colored glass jar with a PFTE lined screw cap. Mix thoroughly by shaking until no lumps are visible, and the silica gel is free flowing and no longer sticks to the side of the jar.
- 7.6.9 Alumina, Neutral - Super I - Scientific Absorbents. Purchase and use only activated alumina. Store in an oven at 130°C when not in use.
- 7.6.9.1 Each new lot of alumina must be tested upon receipt and before use. Elute a solution containing all of the ^{13}C internal standards and native analytes through a column packed with the new lot of alumina. Collect the 5% and 60% fractions together and analyze by HRMS. Archive the 80 mL of hexane in a separate container. The target analytes and internal standard recoveries must be greater than 85% in the final fraction. If the recovery is less than 85% for any compound or internal standard, the ratios and volumes of the elution solvents must be optimized and the test is repeated until all compounds meet the recovery criteria.
- 7.6.10 5% methylene chloride in hexane. Add 15 mL methylene chloride to 285 mL hexane. Store in an amber-colored glass bottle at room temperature until use.
- 7.6.11 65% methylene chloride in hexane - add 390 mL methylene chloride to 210 mL hexane. Store in an amber-colored glass bottle at room temperature until use.
- 7.7 Activated Carbon Cleanup
- 7.7.1 Silica Gel. Refer to section 7.6.6
- 7.7.2 J.T Baker Carbon, Activated Powder, E345-07, or equivalent
- 7.7.3 Thoroughly mix 5% (by weight). Activated J.T Baker carbon and 95% (by weight) Fisher Chromatographic silica gel (100-200 mesh). Activate in an oven at 130°C for 6 hours. Store in a desiccator in an amber colored bottle with a PTFE lined lid until use. Do not label the bottle until oven activation is completed to avoid heat damage to the label.
- 7.7.4 Toluene, pesticide quality or equivalent.
- 7.7.5 Methylene Chloride, pesticide quality or equivalent.

- 7.7.6 Benzene, pesticide quality or equivalent.
- 7.7.7 Methanol, pesticide quality or equivalent.
- 7.7.8 Cyclohexane, pesticide quality or equivalent.
- 7.7.9 Tetradecane, pesticide quality or equivalent.
- 7.7.10 Hexane, pesticide quality or equivalent.
- 7.7.11 Acetone, pesticide quality or equivalent.
- 7.8 Standard and Calibration Solutions: Certified Reference Standards purchased from Cambridge Isotope Laboratories (CIL, Andover Massachusetts), and Wellington Laboratories (Guelph, Ontario, Canada). If the chemical purity is 98% or greater, the weight can be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark at room temperature in screw-capped vials with PTFE-lined caps.
 - 7.8.1 Nonane, pesticide quality or equivalent.
- 7.9 Stock Solutions: Standards are used as received after being sonicated and transferred to 1.0 mL amber glass vials with PTFE lined caps.
 - 7.9.1 Initial Calibration Standards:
 - 7.9.1.1 1613B/8290/8290A: CS1-CS5. CIL Catalog No. EDF-9999. (See Table 5).
 - 7.9.1.2 23/0023A/TO-9A: CS1-CS5. CIL Catalog No. EDF-4052. (See Table 6)
 - 7.9.2 Initial Calibration Verification Standard: Wellington Laboratories Catalog No. EPA-1613-CS3.
 - 7.9.3 Daily Calibration Verification Standards
 - 7.9.3.1 1613B/8290/8290A: CS3. CIL Catalog No. EDF-9999-3. (See Table 7).
 - 7.9.3.2 1613B/8290/8290A: CS3. CIL Catalog No. EDF-4141. (See Table 7).

Note: This standard can be used as both the Continuing Calibration Standard and the DB/Rtx-5 GC Window Defining Mix/Column Performance Check Solution.

 - 7.9.3.3 23/0023A/TO-9A: CS3. CIL Catalog No. EDF-4052-3. (See Table 8)
- 7.9.4 PAR Native Standard Stock Solution: CIL Catalog No. EDF-7999-10x (see Table 11)
400-4000 ng/mL in nonane, 1.2 mL.
- 7.9.5 Internal Standard Stock Solution
 - 7.9.5.1 1613B/: CIL Catalog No. EDF-8999, (see Table 13), 100 ng/mL ($^{13}\text{C}_{12}$ -OCDD 200 ng/mL) in nonane, 500 μL .
 - 7.9.5.2 23/0023A/TO-9A: CIL Catalog No. EDF-4053, (see Table 15), 1000 ng/mL ($^{13}\text{C}_{12}$ -OCDD 2000 ng/mL) in nonane, 1.2 mL.

- 7.9.6 23/0023A/TO-9A Surrogate Standard Stock Solution: CIL Catalog No. EDF-4054, (see Table 16), 1000 ng/mL in nonane, 1.2 mL.
- 7.9.7 Cleanup Standard Stock Solution: CIL Catalog No. ED-907, (see Table 14), 50 µg/mL in nonane, 1.2 mL.
- 7.9.8 ¹³C₁₂-2,3,7,8-TCDD Labeled Standard Stock Solution: CIL Catalog No. ED-900, 50 µg/mL in nonane, 1.2 mL.
- 7.9.9 ¹³C₁₂-2,3,7,8-TCDF Labeled Standard Stock Solution: CIL Catalog No. EF-904 50 µg/mL in nonane, 1.2 mL.
- 7.9.10 ¹³C₁₂-1,2,3,4-TCDD Labeled Standard Stock Solution: CIL Catalog No. ED-911, 50 µg/mL in nonane, 1.2 mL.
- 7.9.11 ¹³C₁₂-1,2,3,7,8,9-HxCDD Labeled Standard Stock Solution: CIL Catalog No. ED-996, 50 µg/mL in nonane, 1.2 mL.
- 7.9.12 PCDD/PCDF Window Defining and Isomer Specificity Mixture: CIL Catalog No. EDF-4147 (see Table 18 and Table 19). This standard is used for qualitative purposes only and is not considered quantitative.

7.10 Secondary Stock Solutions

- 7.10.1 Cleanup Standard Secondary Stock Solution: Dilute 0.100 mL of the stock solution in section 7.9.7 to 1.0 mL in a volumetric flask with nonane to a final concentration of 5.0 µg/mL.
- 7.10.2 Cleanup Standard Working Stock Solution: Dilute 0.120 mL of the stock solution in section 7.10.1 to 3.0mL in a volumetric flask with nonane to a final concentration of 200 ng/mL.
- 7.10.3 ¹³C₁₂ TCDD/TCDF Internal Standard Secondary Stock Solution: Dilute 0.100 mL of the stock solutions in sections 7.9.8 and 7.9.9 to 5 mL in a volumetric flask with nonane to a final concentration of 1000 ng/mL.
- 7.10.4 Recovery Standard (RS) Secondary Stock Solution: Dilute 1.0 mL of the stock solutions in sections 7.9.10 and 7.9.11 to 10 mL in a volumetric flask with nonane to a final concentration of 5.0 µg/mL.
- 7.10.5 Native Standard Working Stock Solution – Dilute 0.300 mL of stock solution in 7.9.4 to 3.0 mL in a volumetric flask with nonane for a final concentration of 40-400 ng/mL.

7.11 Standards and Spiking Solutions

- 7.11.1 PCDD/PCDF Window Defining and Isomer Specificity Standard: Combine 25 µL of the standard solution in section 7.9.12, 5 mL of the IS spiking solution in section 7.11.3, 5 µL of the RS stock solution in section 7.10.4, and 500 µL of nonane in a 10 mL vial. Concentrate the solution to 500 µL with a stream of nitrogen and transfer to 1.0 mL amber glass vials with PTFE lined cap.

- 7.11.2 PAR Native Standard Spiking Solution: Dilute 250 µL of the working stock solution in section 7.10.5 to 50 mL in a graduated cylinder with acetone to a final concentration of 0.2-2.0 ng/mL. 1.0 mL of this solution is added to each IPAR, OPR, LCS or MS/MSD sample. See Table 11 for a complete list of compounds and their concentrations.
- 7.11.3 1613B/ Internal Standard Spiking Solution: Dilute 1000 µL of the stock solution in section 7.9.5.1 to 100 mL in a graduated cylinder with acetone to a final concentration of 1.0 ng/mL (¹³C₁₂-OCDD 2.0 ng/mL). 1.0 mL of this solution is added to each sample, method blank, and QC sample. See Table 13 for a complete list of compounds and their concentrations.
- 7.11.4 23/0023A/TO-9A Internal Standard Spiking Solution: Dilute 100 µL of the stock solution in section 7.9.5.2 to 100 mL in a graduated cylinder with acetone to a final concentration of 1.0 ng/mL (¹³C₁₂-OCDD 2.0 ng/mL). 1.0 mL of this solution is added to each sample, method blank, and QC sample. See Table 13 for a complete list of compounds and their concentrations.
- 7.11.5 23/0023A/TO-9A Surrogate Standard Spiking Solution: Dilute 500 µL of the stock solution in section 7.9.6 to 25 mL in a graduated cylinder with nonane to a final concentration of 20 ng/mL. 100 µL of this solution is added to each sample train components before sampling. See Table 16 for a complete list of compounds and their concentrations.
- 7.11.6 Cleanup Standard Spiking Solution: Dilute 100 µL of the stock solution in section 7.10.2 to 100 mL in a volumetric graduate with hexane to a final concentration of 0.20 ng/mL. 1.0 mL of this solution is added to each sample, method blank, and QC sample extract prior to cleanup. See Table 14 for a complete list of compounds and their concentrations.
- 7.11.7 ¹³C₁₂ TCDD/TCDF Internal Standard Spiking Solution: Dilute 100 µL of the stock solution in section 7.10.3 to 100 mL in a volumetric flask with acetone to a final concentration of 1.0 ng/mL. 1.0 mL of this solution is added to each sample, method blank, and QC sample extract that are extracted for TCDD and/or TCDF analysis only.
- 7.11.8 Recovery Standard Spiking Solution: Add 10 mL of nonane to a 12 mL amber vial with a Class A glass pipette. With a pipettor remove 200 µL of nonane from the vial and add 200 µL of the stock solution in section 7.10.4 to a final concentration of 0.1 µg/mL. 20 µL of this solution is added to each sample, method blank, and QC sample extract.
- 7.12 Stability of Solutions: Standards have an expiration of ten (10) years from date of receipt unless otherwise specified by the manufacturer. Standard solutions used for quantitative purposes must be analyzed periodically, and must be assayed against reference standards before use.
- 7.13 Perfluorokerosene (PFK) is used in neat form to tune and calibrate the mass spectrometer. Fluka (Catalog No. - 77275) has been found to be superior to other sources of PFK.

8. Sample Collection, Preservation and Storage

- 8.1 Sampling is not performed for this method by TestAmerica Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, Receipt and Log In of Commercial

Samples, current revision. Sample container and preservation recommendations are listed in the table below.

Sample Holding Times, Containers, and Preservation

Method:	1613B	8290/8290A ¹	23	0023A ¹	TO-9A
Holding Times	Samples – 1 year from collection to extraction Extracts – 1 year from extraction to analysis	Samples – 30 days from collection to extraction Extracts – 45 days from extraction to analysis Tissue Extracts – 45 days from collection to analysis	Samples – 30 days from collection to extraction Extracts – 45 days from extraction to analysis	Samples – 30 days from collection to extraction Extracts – 45 days from extraction to analysis	Samples – 7 days from collection to extraction Extracts – 40 days from extraction to analysis
Containers	Amber Glass	Amber Glass	See KNOX-ID-0012	See KNOX-ID-0012	See KNOX-ID-0012
Preservation:					
Aqueous Samples	0-4 °C in the dark If residual chlorine is present, add 80 mg/L sodium thiosulfate. If pH > 9, adjust to pH 7-9 with sulfuric acid	4 °C ± 2 °C in the dark	N/A	N/A	N/A
Solid Samples	<-10 °C in the dark	4 °C ± 2 °C in the dark	N/A	N/A	N/A
Tissue Samples	<-10 °C in the dark	<-20 °C in the dark ²	N/A	N/A	N/A
Air Samples	N/A	4 °C ± 2 °C in the dark	4 °C ± 2 °C in the dark	4 °C ± 2 °C in the dark	≤4 °C in the dark

Note:

- For methods 8290, 8290A and 0023A the holding times listed are recommendations. PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed can be as high as a year for certain matrices. The results of samples analyzed after the holding time expiration date must be considered to be minimum concentrations and must be identified as such in the final report. Sample extracts, however, must always be analyzed within 45 days of extraction. (For the State of South Carolina and the New Jersey DEP – The holding times are as listed in the table and are not considered recommendations.)
- If the freezer used to store samples is not capable of reaching a temperature of <-20 °C when the temperature control is set to its maximum limit, a storage higher temperature is acceptable as long as it is <-10 °C.

9. Quality Control

9.1 Initial Demonstration of Capability

- 9.1.1 Initial precision and recovery (IPR) samples are analyzed to demonstrate the ability to generate acceptable precision and accuracy.
- 9.1.2 For aqueous samples, extract, concentrate, and analyze four 1-L aliquots of reagent water spiked with labeled internal standards and the precision and recovery standard according to the procedures in section 11. For non-aqueous samples, extract, concentrate, and analyze four aliquots of sand or sodium sulfate spiked with labeled

internal standards and the precision and recovery standard according to the procedures in section 11. It is recommended that a method blank be prepared with the IPR samples.

- 9.2 Extracts must be stored in the dark at room temperature in amber or clear glass vials prior to analysis.
- 9.2.1 Using the results of the set of four analyses, compute the average concentration (X) of the extracts in ng/mL and the standard deviation of the concentration(s) in ng/mL for each compound.
- 9.2.2 For each compound, compare s and X with the corresponding limits for initial precision and recovery in Table 9 for method 1613B and Table 10 for methods 8290, 8290A 23, 0023A, and TO-9A. If s and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples can begin. If, however, any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, system performance is unacceptable for that compound. Correct the problem and repeat the test.
- 9.3 The method detection limit (MDL) study described in Section 13 must be completed with acceptable results before analysis of samples can begin.
- 9.4 A laboratory method blank must be run along with each analytical batch of 20 (10, including field blank if provided, for TO-9A) or fewer samples. The method blank is normally analyzed immediately after the calibration standards. The method blank consists of reagent water for aqueous samples, and a clean solid matrix (sand or sodium sulfate) for solid samples. The method blank extracts must be subject to the same clean up procedures as the associated sample extracts. The method blank must meet the following acceptance criteria;
- The concentration of target analytes in the method blank must be less than the MDL. If the concentration of target analytes in the method blank is greater than the MDL but less than the minimum level (ML), corrective action is required but the associated samples can be reported. At a minimum, corrective must include the addition of "B" qualifiers to all associated samples with analytes detected in the method blank above the MDL.
 - If the concentration of target analytes in the method blank is greater than minimum level (ML) but less than 5% of the concentration in the associated samples, corrective action is required but the associated data can be reported. At a minimum, corrective action must include the addition of "B" qualifiers to all associated samples with analytes detected in the method blank above the ML and documentation in the case narrative. (Note: Method Blank values must be less than the ML for Ohio VAP samples)
 - If the method blank sample fails to meet the acceptance criteria, the Project Manager is notified and the entire sample batch is re-extracted. If there is insufficient sample volume remaining for re-extraction, the client is contacted for information about the availability of additional sample volume. If there is no additional sample available, the original sample data is flagged and reported. A nonconformance memo is initiated describing the problem and corrective action. The problem and corrective action is documented in the project narrative.

- If there is no target analyte greater than the minimum levels (ML) in the samples associated with an unacceptable method blank, the data can be reported with qualifiers. Such action must be done in consultation with the client.

9.4.1 The method blank internal standard recoveries must be within the established control limits. If internal standard recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If internal standard recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples is required if the method blank does not demonstrate that the analysis is free of contamination. If the method blank internal standard recoveries are outside the QC limits and the decision is made to report the sample results, an NCM must be initiated and the reason for accepting the sample results clearly documented. Consultation with the client before acceptance must take place.

9.4.2 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," an NCM is initiated and appropriate comments made in the report narrative to provide further documentation.

9.4.3 Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.5 Instrument Blank

9.5.1 Instruments must be evaluated for contamination during each 12 hour analytical run. This is accomplished by analysis of a method blank if available. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of solvent with the internal standards and recovery standards added. It is evaluated in the same way as the method blank.

9.6 Laboratory Control Sample (LCS) or Ongoing Precision and Recovery (OPR)

9.6.1 An LCS/OPR sample is analyzed along with each analytical batch of 20 (10, including field blank if provided, for TO-9A) or fewer samples. LCS/OPR spike components, concentrations, and control limits are given in Table 11.

9.6.2 If any analyte in the LCS is outside the control limits, corrective action must occur. Corrective action includes:

- If the LCS/OPR sample fails to meet the acceptance criteria, the Project Manager is notified and the entire sample batch is re-extracted. If there is insufficient sample volume remaining for re-extraction, the client is contacted for information about the availability of additional sample volume. If there is no additional sample available, the original sample data is flagged and reported. A nonconformance memo is initiated describing the problem and corrective action. The problem and corrective action is documented in the project narrative.
- If the batch is not re-extracted and reanalyzed, an NCM must be initiated and the reasons for accepting the batch must be clearly presented in the project records and the

report. (An example of an acceptable reason for not reanalyzing might be that the matrix spike and matrix spike duplicate recoveries are within control limits, the method blank and sample internal standard recoveries are within limits, and the data clearly demonstrates that the problem was confined to the LCS/OPR).

- For method TO-9A calculate the precision (%D) relative to the previous TO-9A LCS. The precision must be within $\pm 30\%$.

9.6.3 Ongoing monitoring of the LCS/OPR provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.7 Internal Standards.

Internal standards are spiked into all samples, blanks, and laboratory control samples to assess method performance on the sample matrix. The recovery of each labeled internal standard must be within the limits in Table 13 for methods 1613B, 8290 and 8290A or in Table 15 for methods 23, 0023A, and TO-9A.

9.7.1 If the recovery is outside these limits the following corrective action must be taken:

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- If the recovery of any internal standard is less than the lower control limit, calculate the S/N ratio of the internal standard. If the S/N is > 10 and the estimated detection limits (EDLs) are less than the minimum levels (ML's), report the data as is with qualifiers in the report and a discussion in the case narrative. If the S/N is < 10 or the estimated detection limits (EDLs) are greater than the minimum levels (ML's), re-extract and re-analyze the sample. If the poor internal standard recovery is judged to be a result of sample matrix, a reduced portion of the sample can be re-extracted or additional clean-ups can be employed. The decision to reanalyze or flag the data is made in consultation with the client.

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Method 8290 only.

When method 8290 is performed a matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every 20 samples of a given matrix. Note that a MS/MSD is not required for Method 8290A. The MS/MSD is spiked with the same subset of analytes as the LCS (See Table 12). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action is to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis can proceed. The reasons for accepting the batch must be documented in the report narrative.

- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the analysis is out of control and corrective action must be taken. Corrective action normally includes reparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCSD must be analyzed. The LCSD is evaluated using the same acceptance criteria as the LCS. The RPD of the LCS and LCSD are compared to the acceptance limits in Table 12.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds are diluted out.

9.9 Surrogate Standards – Methods 23, 0023A, TO-9A

Field surrogate standards are added to the collection media prior to sample collection when performing methods 23, 0023A, or TO-9A. The surrogate recoveries are calculated relative to the internal standards and are a measure of sampling efficiency. The recovery of the surrogate standards must be within the limits specified in Table 16. Poor recoveries of the surrogate standards can indicate breakthrough in the sampling train.

9.9.1 If the recovery is outside these limits the following corrective action must be taken:

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Flag the results that are outside control limits and notify the Project Manager. The client must be notified and consulted for additional corrective action.

9.10 For Ohio VAP, the laboratory must take corrective action for any QC outliers which includes re-extraction if there is sufficient holding time and sample volume. Also, the client may not make decisions regarding whether or not the laboratory takes corrective action.

10. Calibration and Standardization

10.1.1 Two types of calibration procedures are required. One type, initial calibration, is required before any samples are analyzed and is required intermittently throughout sample analyses as dictated by the results of continuing calibration procedures described below. The other type, continuing calibration, consists of analyzing the column performance check solution and a calibration solution (CS3). No samples are to be analyzed until acceptable calibration as described in sections 10.2 and 10.2.8.1 is demonstrated and documented. A 2uL injection volume is specified for all extracts, blanks, calibration solutions and performance check samples. A 1uL injection volume can be used; however, the laboratory must keep the injection volume the same throughout calibration and analysis.

10.2 Initial Calibration

- 10.2.1 Prepare multi-level calibration standards containing the compounds and concentrations as specified in Table 5 for methods 1613B, and 8290/8290A or in Table 6 for methods 23, 0023A, or TO-9A. Store calibration standards at room temperature in the dark. Calibration standard solutions have an expiration date of ten (10) years from date of receipt unless otherwise specified by the manufacturer/supplier.
- 10.2.2 Establish operating parameters for the GC/MS system (suggested operating conditions are displayed in Figure 1 and Figure 2). For method 1613B adjust the GC conditions to meet the relative retention times for the PCDDs/PCDFs listed in Table 3. The cycle time for MID descriptors must be ≤ 1 sec.
- 10.2.3 By using a PFK molecular leak, tune the instrument to meet the minimum resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to the m/z 303.9016 (from TCDF). By using peak matching conditions and the aforementioned PFK reference peak, verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Document that the resolving power at reduced accelerating voltage of m/z 380.9760 is greater than 10,000 (10 percent valley).
- 10.2.4 Analyze 2 μ L of the Window Defining Mixture and set the switchpoints for the MID descriptors. The switchpoints must be set to encompass the retention time window of each congener group.
- 10.2.5 If the initial calibration is being performed on the DB-5 or RTX-5 column, analyze 2 μ L of the Column Performance solution or Mixture Solution. The chromatographic peak separation between 2,3,7,8-TCDD and the closest eluting non-2,3,7,8-TCDD isomer must be resolved with a % Valley of ≤ 25 , where

$$\% \text{ Valley} = \frac{\text{baseline to valley height of closest eluting isomer}}{\text{peak height of 2,3,7,8 - TCDD}} \times 100$$

If the initial calibration is being performed on the DB-225 or RTX-225 column, analyze 2 μ L of the TCDF Column Performance solution. The chromatographic peak separation between 2,3,7,8-TCDF and the closest eluting non-2,3,7,8-TCDF isomer must be resolved with a % Valley of ≤ 25 , where

$$\% \text{ Valley} = \frac{\text{baseline to valley height of closest eluting isomer}}{\text{peak height of 2,3,7,8 - TCDF}} \times 100$$

- 10.2.6 Analyze 2 μ L of each of the five calibration standards and calculate the RRF of each analyte vs. the appropriate internal standard listed in Table 3 for methods 1613B, 8290/8290A or in Table 4 for methods 23, 0023A, and TO-9A using the following equation;

$$\text{RRF} = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

As = sum of the areas of the quantitation ions of the compound of interest
 Ais = sum of the areas of the quantitation ions of the appropriate internal standard
 Cis = concentration of the appropriate internal standard
 Cs = concentration of the compound of interest

- 10.2.6.1 Calculate the mean relative response factor (mean RRF) and the percent relative standard deviation (RSD) of the relative response factors for each compound of interest in the five calibration standard solutions using the following equations;

$$\overline{\text{RRF}}_{n=5} = \frac{1}{n} \times \sum_{i=1}^n \text{RF}_i$$

$$\text{RSD}_{n=5} = \sqrt{\frac{\sum_{i=1}^n (\text{RF}_i - \overline{\text{RF}})^2}{n-1}} \times \frac{100}{\overline{\text{RRF}}}$$

- 10.2.7 Criteria for Acceptable Calibration - The criteria listed below for acceptable calibration must be met for each initial calibration standard before sample analyses are performed. If acceptable initial calibration is not achieved, identify the root cause, perform corrective action, and repeat the initial calibration. If the root cause can be traced to problems with an individual analysis within the calibration series, follow the procedure in Test America Policy CA-T-P-002 Selection of Calibration Points, current revision (see reference section 16.8).
- 10.2.7.1 The percent relative standard deviation (RSD) for the mean relative response factors must be within the acceptance criteria listed in Table 5 for methods 1613B, 8290/8290A, or in Table 6 for methods 23, 0023A, and TO-9A.
- 10.2.7.2 The peaks representing the PCDDs/PCDFs and labeled compounds in the calibration standards must have signal-to-noise ratios (S/N) ≥ 10 .
- 10.2.7.3 The ion abundance ratios must be within the specified control limits in Table 22.
- 10.2.7.4 For method 1613B the absolute retention time of $^{13}\text{C}_{12}$ -1234-TCDD must exceed 25.0 minutes on the DB/Rtx-5 column and 15.0 minutes on the DB/Rtx-225 column.
- 10.2.7.5 Corrective action can include replacing the injector port liner, replacing the injector port septum, removal of a small portion of the front of the analytical column, replacing the autosample syringes and rinse solvent, adjusting the instrument tuning, cleaning the ion volume or ion source, installing a new analytical column, and replacing the calibration standard solutions.
- 10.2.8 Analyze 2 μL of the Initial Calibration Verification (ICV) Standard in section 7.9.2 after the completion of the initial calibration prior to sample analysis. Calculate the concentration of the ICV using the RRF's from the CS3 standard analyzed in section 10.2.6 and the formula in section 12.3.4. Calculate the percent difference (%D)

between the expected and the calculated ICV concentration using the following formula.

$$\%D = \frac{(C_{Exp} - C_{Calc})}{C_{Exp}} \times 100$$

Where:

C_{Exp} = The expected concentration of the Standard.

C_{Calc} = The calculated concentration of the Standard.

- 10.2.8.1 The general criteria for percent difference acceptance limits is less than or equal to $\pm 25\%$ for all native compounds. The warning limits for percent difference is $\pm 25 - 35\%$.
- 10.2.8.2 All data associated with compounds with percent differences in the warning limits must be reviewed before acceptance.
- 10.2.8.3 All data associated with compounds with percent differences outside the warning limits must be documented as an NCM. Corrective action must be taken and can include the following
 - Reanalyze the ICV Standard
 - Replace and reanalyze the ICV Standard
 - Evaluate the instrument performance
 - Evaluate the Initial Calibration Standards

10.3 Continuing Calibration

- 10.3.1 Continuing calibration is performed at the beginning of a 12 hour period after successful mass resolution and GC resolution performance checks. A calibration check is also required at the end of a 12 hour period when performing method 8290/8290A or 0023A.
- 10.3.2 Document the mass resolution performance as specified in section 10.2.3. The mass resolution checks must be performed at the beginning and at the end of each 12-hour shift.
- 10.3.3 Analyze 2 μ L of the Window Defining Mixture and or Column Performance Solution Mixture under the same instrument conditions used to perform the initial calibration. Determine and document acceptable column performance as described in section 10.2.4 and 10.2.5.
- 10.3.4 Analyze 2 μ L of the Daily Calibration Standard Solution (CS3). Calculate the concentrations and percent difference of the standard using the formulas in sections 12.3.4 and 10.2.7.5.

Note: The combined Continuing Calibration Standard/Window Defining Mix/Column Performance Solution specified in section 7.9.3.2 can be used in section 10.3.2, 10.3.4, and 10.3.6.

- 10.3.5 Criteria for Acceptable Calibration - The criteria listed below for acceptable calibration must be met at the beginning of each 12 hour period that samples are analyzed. If acceptable beginning continuing calibration criteria is not met, identify the root cause, perform corrective action and repeat the continuing calibration. If the second consecutive beginning continuing calibration does not meet acceptance criteria, additional corrective action must be performed. Acceptable performance must be demonstrated after two consecutive failing beginning continuing calibrations by the analysis of two consecutive acceptable beginning continuing calibrations or by analysis of a new initial calibration.
- 10.3.5.1 The measured concentration or percent difference for each compound must be within the acceptance criteria limits in Table 7 for methods 1613B, 8290/8290A or in Table 8 for methods 23, 0023A, and TO-9A.
 - 10.3.5.2 For method 1613B the relative retention times of PCDDs/PCDFs and labeled compounds in the standard must be within the limits in Table 3.
 - 10.3.5.3 The peaks representing the PCDDs/PCDFs and labeled compounds in the calibration standard must have signal-to-noise ratios (S/N) ≥ 10 .
 - 10.3.5.4 The ion abundance ratios must be within the specified control limits in Table 22.
 - 10.3.5.5 Corrective action can include all of the items specified in section 10.2.7.5.
 - 10.3.5.6 When performing method 8290/8290A, or 0023A, if the continuing calibration fails at the beginning of a 12-hour shift, the instructions in section 10.3.5 must be followed. If the continuing calibration check performed at the end of a 12 hour period fails by no more than ± 25 percent RPD for unlabeled native analytes and ± 35 percent RPD for labeled standards, the closing standard must not be used as a beginning calibration standard for the next 12-hour shift and the requirements in section 10.3.5 must be met before analysis can continue. Use the mean RRF from the two daily continuing calibration runs to compute the analyte concentrations, instead of the RRFs obtained from the initial calibration. If the continuing calibration check performed at the end of a 12 hour period fails by more than ± 25 percent RPD for unlabeled native analytes and ± 35 percent RPD for labeled standards initiate corrective action and reanalyze all sample extracts analyzed during the 12 hour period encompassing the failed end of shift calibration check.

It is realized that it might not always be possible to achieve all RF criteria. For example, the RF criteria for 13C12-HpCDD and 13C12-OCDD were not met, however the RF values for the corresponding unlabeled compounds were within the criteria established in this procedure. The data quality for the unlabeled HpCDD and OCDD values were not compromised as a result of the calibration event. In these situations, the analyst must consult with the group manager and the project manager to assess the impact on the data quality objectives on the affected samples. Corrective action must be taken

and any decision to report sample data in this situation must be made in conjunction with the client. An NCM must be initiated if the data is to be reported.

- 10.3.6 Daily calibration must be performed every 12 hours of instrument operation. The 12 hour shift begins with the documentation of the mass resolution followed by the injection of the Window Defining Mixture or Column Performance Solution Mixture and the Daily Calibration Standard.
 - 10.3.6.1 For methods 1613B, 23, TO-9A- The mass resolution documentation must also be performed at the end of the 12 hour shift. If the lab is operating consecutive 12 hour shifts, the mass resolution check from the end of the previous period can be used for the beginning of the next period.
 - 10.3.6.2 For method 8290/8290A or 0023A - The Continuing Calibration Standard check and mass resolution documentation must also be performed at the end of the 12 hour shift. If the lab is operating consecutive 12-hour shifts, the Window Defining Mixture and/or Column Performance Solution Mixture must be analyzed at the beginning of each 12-hour period. The mass resolution and continuing calibration checks from the previous period can be used for the beginning of the next period.

11. Procedure

- 11.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variations in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variations in the procedure, except those specified by project specific instructions, must be completely documented using a Nonconformance Memo and approved by a Technical Specialist, Project Manager, and QA Manager. If contractually required, the client must be notified.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.
- 11.2 Screening process for samples with high concentration of Dioxins/Furans.
 - 11.2.1 Samples received are carefully reviewed before starting the extraction process. Any samples that are received from known Dioxin/Furan sites and samples that contain keywords such as PCP (pentachlorophenol) site, wood treaters, PCB sites, and fire/burn sites can be subjected to the screening process.
 - 11.2.2 Screening is done as a precaution to minimize the chance of having high level samples exposing the preparation and analytical areas to excessive amounts of Dioxin/Furan's, thereby potentially contaminating areas and other samples contained in those areas. See Figure Attachment I for LRMS Dioxin Screen Strategy.
 - 11.2.3 Mix Sample well, weigh out 2.5 g and place in a 40 mL vial.
 - 11.2.4 Add 10 mL of toluene and shake on a shaker table for 3 hours.

- 11.2.5 After contents have settled, remove the toluene from the vial and place in a fresh, clean vial containing 100 μ L of tetradecane as a keeper. Rinse vial 3 times with 1 mL of toluene to insure complete transfer of extract.
- 11.2.6 Solvent exchange the extract by placing it on the nitrogen concentration device. Concentrate the extract to near dryness. Add 4 mL of hexane and concentrate to near dryness again. Repeat again and then bring the extract to volume with 2 mL of hexane.
- 11.2.7 Run the extract thru a silica gel/alumina column cleanup as detailed in section 11.9.3. After the silica gel/alumina column cleanup is completed, put the extract back on nitrogen concentration device and concentrate to approximately 0.5 mL.
- 11.2.8 Transfer the concentrated extract to a 2 mL, clear, crimp top vial marked at 1.0 mL. Rinse the 40 mL vial several times with small amounts of hexane to complete the transfer. Adjust volume of the extract to 1.0 mL. Take extract to GC/MS group for analysis.

11.3 Sample Pretreatment

11.3.1 Tissue Samples

- 11.3.1.1 If the sample matrix is tissue and has not been homogenized prior to sample receipt, the entire sample is blended to provide a homogeneous sample. At least 20 g of tissue is homogenized if possible to allow for reanalysis if necessary.
- 11.3.1.2 Cut tissue into pieces of a uniform size (approximately 1 inch square). Homogenize the tissue sample in a laboratory blender.
- 11.3.1.3 Weigh out 10 grams of the homogenized tissue sample, record the sample weight on the sample prep sheet or in a logbook. Add the sample along with 20 g of sodium sulfate to a laboratory blender. Blend the tissue/sodium sulfate mixture, while adding small chips of dry ice as necessary, to achieve a powder like consistency. Additional amounts of sodium sulfate can be added to achieve a powder like consistency.

If a Lipids determination has been requested see Appendix I

11.3.2 Fly Ash Samples

- 11.3.2.1 If the sample matrix is fly ash and is to be analyzed by method 8290, pretreat the sample with HCl as follows:
- 11.3.2.2 Weigh 10 ± 0.05 g of the fly ash sample and transfer to a 240 mL glass jar. Record the sample weight on the sample prep sheet. If a sample is designated for MS/MSD analysis, prepare two additional portions of the sample and label them as the MS and MSD samples.

- 11.3.2.3 Add 150 mL of 1N HCl to the sample. Seal the jar with a PTFE lined screw cap and shake for 3 hours at room temperature.
- 11.3.2.4 Rinse a glass fiber filter with reagent water, carefully fit the glass fiber filter into a Buchner funnel and filter the sample through the glass fiber filter, placed in the Buchner funnel attached to a 1 L vacuum flask. Rinse the sample bottle twice with small amounts of reagent water, making sure that all particulate matter is transferred onto the glass fiber filter. Wash the fly ash cake with approximately 500 mL reagent water.
- 11.3.2.5 Add 1.0 mL of the internal standard spiking solution (see section 7.11.3) to the sample. Record the standard solution ID and volume spiked on the sample prep sheet. Initial and date the entry. Extract the sample and glass fiber filter by Dean-Stark Soxhlet extraction in section 11.5.

11.4 Aqueous Sample Extraction

- 11.4.1 Remove samples from the refrigerator and allow them to come to room temperature before measuring the volume or performing the extraction. Inspect the samples carefully for biphasic sample characteristics. If this condition exists, document the observation, contact project manager for instructions before proceeding with the extraction.
- 11.4.2 For aqueous samples that contain visible solids, a percent solid determination must be performed using the following procedure.

Add 10 mL of the well shaken sample to a pre-weighed aluminum weighing dish. Weigh the dish to three significant figures. Dry the dish overnight in an oven at 105 °C. Reweigh the dish and calculate the percent solids using the following equation.

$$\%solids = \frac{\text{weight of dish plus sample after drying} - \text{weight of dish}}{\text{weight of dish plus sample before drying} - \text{weight of dish}} \times 100$$

- 11.4.3 8290 aqueous extraction and analysis: For samples with $\leq 1\%$ solids follow normal extraction procedure. For sample with $\geq 1\%$, consult project manager for further instruction before proceeding with the extraction.

Note: Samples can be filtered, and extracted as a solid or aqueous separately or both fractions of solid and aqueous can also be combined after extraction at client's request.

- 11.4.4 1613 aqueous extraction and analysis: For samples with $\leq 1\%$ solids, samples must be filtered and extracted as solid and aqueous fractions. The extracts from each fraction are then combined into one extract. See Section 11.4.9.1 for filtration. For samples with $> 1\%$, consult project manager for further instructions before proceeding with extraction.
- 11.4.5 Refer to Knoxville SOP, KNOX-QA-0002, current revision, for information on glassware cleaning procedures for extraction glassware. Visually inspect all glassware prior to use for scratches or cracks. Retire and replace any glassware found to be damaged.

- 11.4.6 Place separatory funnels, one for each sample, the method blank, and the OPR, in the positions in the rotary extractor.
- 11.4.7 Place a 600 mL concentration tube directly beneath each separatory funnel in the tube holder.
- 11.4.8 Plug a glass funnel with glass wool and pour in some sodium sulfate (about 1 to 2 inches from the top). Rinse the sodium sulfate with methylene chloride. After the funnel stops dripping, place the funnel on top of the concentrator tube.
- 11.4.9 If solids are not observed in the sample, mark the level of the sample on the sample bottle in order to measure the volume later and carefully add the sample to the separatory funnel, taking care not to spill any sample. Using a Class A 1000 mL graduated cylinder measure out 1000 mL of reagent water and add to the separatory funnels marked for the method blank, LCS/OPR, and LCSD (if required).

11.4.9.1 Sample Filtration

- 11.4.9.1.1 Assemble a Buchner funnel with a rubber stopper on top of a clean vacuum filter flask. Insert a 15 cm diameter, 2.7 μ m particle retention glass fiber filter into the funnel. Wet the filter paper with a few mLs of reagent water and apply vacuum to the filter flask.
 - 11.4.9.1.2 Apply vacuum to the flask, mark the level of the sample on the sample bottle in order to measure the volume later and carefully add the sample to the Buchner funnel, swirling the sample remaining in the bottle to suspend any particles.
 - 11.4.9.1.3 Rinse the sample bottle twice with approximately 10 mL portions of reagent water to transfer any remaining particles onto the filter. Rinse any particles off the sides of the Buchner funnel with small quantities of reagent water.
 - 11.4.9.1.4 If the percent solids are $\leq 1\%$, extract the filtrate in a separatory funnel by proceeding to section 11.4.10. Extract the solids on the filter and the filter itself following the procedure in section 11.5.
Do not add internal standards to this portion of the sample, only add internal standards to the aqueous portion of the sample! The resulting extract is combined with the extract of the aqueous portion during the macro concentration step in section 11.8.
- 11.4.10 Using a Class A 1 mL volumetric pipet, add 1 mL of the ^{13}C labeled internal standard spiking solution, as specified in section 7.11.3, to each sample, the method blank, LCS/OPR, LCSD (as required), and MS/MSD (as required) samples. Record the amount of spike used and the spike solution number in the standards logbook and on the benchsheet.

- 11.4.11 Using a Class A 1 mL volumetric pipet, add 1 mL of the PAR native spiking solution, as specified in section 7.11.2, to the designated LCS/OPR, LCSD (as required), and MS/MSD (as required) samples. Record the amount of spike used and the spike solution number in the standards logbook and on the benchsheet.

Note: If the volume of standard in the stock container is less than 10 mL after use, discard the remaining portion and prepare a new batch as specified in section 7.11.2.

- 11.4.12 Add 60 mL of methylene chloride to the sample bottle and shake. Then carefully pour the methylene chloride into the separatory funnel. Add 60 mL of methylene chloride to the method blank, LCS/OPR, and LCSD (if required) as well.
- 11.4.13 Secure the separatory funnel with the rotator retaining straps and rotate for 2 minutes.

CAUTION: Care must be used while performing this operation. Vent the separatory funnel frequently. Goggles can be worn when performing this procedure.

- 11.4.14 Allow the water and the methylene chloride to separate for 10 minutes. If it is not separated after 10 minutes, try to break up the emulsion by gently swirling the sample or tilting the separatory funnel on its side.
- 11.4.15 Drain the methylene chloride from the separatory funnel into the glass funnel that is filled with sodium sulfate, allowing the extract to drip into the concentrator tube. Be careful not to allow water to escape the separatory funnel or the sodium sulfate will harden and block the flow of the extract. When an emulsion is present, do not drain the emulsion until the third methylene chloride shake has been completed. If at least 10 minutes has elapsed and other ways of breaking up or reducing the size of the emulsion have failed the following steps can be tried to reduce the impact of the emulsion on the sodium sulfate.
- 11.4.15.1 Place a large piece of pre-cleaned glass wool in the funnel containing the sodium sulfate.
- 11.4.15.2 Spread the glass wool out, covering the entire surface of the sodium sulfate to about a depth of about 5 to 10 mm. If the emulsion is hard to break up and persistent, a small, additional layer of sodium sulfate is added on top of the glass wool.
- 11.4.15.3 Drain the solvent and emulsion layer into the funnel being careful to drain no more than 60 mL of volume if a clear phase layer cannot be determined.
- 11.4.15.4 If this procedure is used the funnel is rinsed with an extra 30 mL of methylene chloride to ensure all analytes are rinsed into the concentrator tube after the third portion of methylene chloride has drained through the sodium sulfate in section 11.4.17.

- 11.4.16 Repeat steps 11.4.12 through 11.4.15 two more times.

- 11.4.17 After the third methylene chloride portion has filtered through the sodium sulfate, rinse the funnel with approximately 40 mL of methylene chloride.

- 11.4.18 Remove the separatory funnel from the hood and pour the extracted water into the extracted water waste's carboy.
- 11.4.19 Fill the empty sample bottle to the marked level with tap water. Pour the tap water into a Class A 1000 mL graduated cylinder. Record the volume of sample used on the benchsheet.
- 11.4.20 Proceed to Macro Extract Concentration by Rapid-Vap in section 11.8.

11.5 Soxhlet Extraction

- 11.5.1 Prepare and label the required number of Soxhlet systems., A Dean-Stark extractor is used to remove the water from all sample matrices except tissue. The Dean-Stark apparatus is installed between the Soxhlet body and the condenser when the components are assembled.
- 11.5.2 The Soxhlet is prepared by cleaning and rinsing per section 6.1.
- 11.5.3 For 1613 solids and for 8290 or 8290A solids that have been designated as sediments: Calculate the amount of solids to be weighed out to achieve a 10 g dry weight of sample. See Attachment II for example of moisture spreadsheet.
 - 11.5.3.1 Batch samples in Quantims and access an EXCEL template to determine the amount of solids to weigh out to achieve a 10 g "dry weight" sample. In EXCEL, select General Template file menu on the right, then select Knx OrgPrep, and then pick Sediment Extraction Amounts, Current Revision; and follow the instructions as they appear. After the information has been acquired, click on the 10g tab and the amounts needed to use appears.
- 11.5.4 Transfer 10 ± 0.05 g of the solid sample (wet weight) into a glass fiber extraction thimble or glass fiber filter paper and put the thimble or filter inside the Soxhlet. Enter the adjusted amount of sample size if adjusting the amount extracted to achieve a 10 g dry weight. If tissue samples are being extracted, add the entire sample and sodium sulfate mixture prepared in section 11.3.1.3. Record the sample weight on the sample prep sheet. Initial and date the entry. Enter the balance ID number on the sample prep sheet. If a sample is designated for MS/MSD analysis, prepare two additional portions of the sample and label them as the MS and MSD samples.

Note: The MS and MSD samples must be prepared at the same weight as the OS to avoid calculation errors in the RPD values.

- 11.5.4.1 For the method blank, LCS/OPR and LCSD (if required) add 10 ± 0.05 g of sodium sulfate to a glass fiber extraction thimble.
- 11.5.4.2 If the matrix is tissue samples, sodium sulfate and dry ice are used for method blank, LCS/OPR, and LCSD (if required). Transfer 20 ± 0.5 g of the sodium sulfate and several small chips of dry ice into an extraction thimble.
- 11.5.4.3 Record the blank matrix type and lot number on the bench sheet.

- 11.5.5 Pour approximately 350 mL toluene into a 500 mL round bottom flask. For tissue samples, use methylene chloride as the extraction solvent. Place the flask in the heating mantle. Add approximately 10 boiling beads and several PTFE boiling chips.
- 11.5.6 Place the extraction thimble in the glass Soxhlet extractor.
- 11.5.7 Assemble the Soxhlet system and secure to the lab supports.
- 11.5.7.1 Place the method blank and QC samples in random positions within the available prep positions in the hood (i.e., do not use the same positions each time for the method blank and QC samples).
- 11.5.8 Spike each sample with 1.0 ml of the internal standard spiking solution (see section 7.11.3) and add a small amount of glass wool if needed to secure the sample material to the top of the extraction thimble. Record the standard solution ID and volume spiked on the sample prep sheet. Initial and date the entry.
- 11.5.8.1 Spike the LCS/OPR, LCSD (if required), and MS/MSD samples with 1.0 ml of the PAR native spiking solutions (see section 7.11.2) prior to adding the glass wool. Record the standard solution ID and volume spiked on the sample prep sheet. Initial and date the entry.
- Note: If the volume of standard in the stock container is less than 10 mL after use, discard the remaining portion and prepare a new batch as specified in section 7.11.2.
- 11.5.9 Adjust the temperature of the heating mantle to bring the solvent in the round bottom flask to a rolling boil. There must be a steady drip from the condensers so that the solvent completely cycles at least 5 times an hour. Record the date and time that the Soxhlet extraction was started on the benchsheet and initial and date.
- 11.5.10 Soxhlet extract the sample in the above manner for a minimum of 16 hours. At the end of the extraction period, turn off the heating mantles. Record the date and time that the Soxhlet extraction was completed on the benchsheet and initial and date.
- 11.5.11 Remove the condensers. If a Dean Stark condenser is used, drain the water from the Dean Stark (the bottom layer of liquid). Then drain the remaining liquid into the soxhlet. Empty the Soxhlet extractor chamber into the round bottom flask and remove the Soxhlet extractor from the 500 mL round bottom flask.
- 11.5.12 Add several (2-3) fresh boiling chips to the flask. Insert a three-ball macro Snyder column into the top of the 500 mL round bottom flask.
- 11.5.13 Apply heat to the 500 mL flask in the heating mantle and reduce the extract volume to approximately 10-15 mL.
- 11.5.14 Transfer the extract into a 40 mL vial containing 100 uL of tetradecane, rinsing the 500 mL flask 3 times with 3 mL of the extraction solvent. Add the rinsings to the 40 mL vial.

- 11.5.15 Place the 40 mL vials into the nitrogen concentration device and reduce the volume to near dryness. Add 4 mL of hexane and swirl the vial. Reduce the volume of hexane to near dryness again to complete the solvent exchange. If the sample exhibits poor solubility in hexane, add approximately 1 mL of benzene with a pipet to the vial to aid in dissolving the residue. Adjust the final volume of the extract with hexane to 12 mL for acid-base cleanup or 2 mL for column cleanup. Proceed to sample cleanup in section 11.9.

11.6 Waste Dilution

- 11.6.1 Organic wastes, oil, solids that dissolve in solvent, and non-aqueous sludge samples can be prepared by the waste dilution technique.
- 11.6.2 Tare a clean 40 mL VOA vial on a laboratory balance. Add an appropriate amount of sample (e.g., 1.0 g) to the VOA vial. If a sample is designated for MS/MSD analysis, prepare two additional portions of the sample and label them as the MS and MSD samples. Prepare method blank, LCS/OPR, and LCSD (if required) samples by adding 12 mL of hexane to a 40 mL VOA vial.
- 11.6.3 Record the weights and volumes used on the laboratory bench sheets and initial and date.
- 11.6.4 Add 1.0 ml of the internal standard spiking solution (see section 7.11.3) to the samples, method blanks, and QC samples. Record the spike solution number and the volume spiked on the sample prep sheet. Initial and date the entry. Add hexane to bring the volume to 12 mL. If the sample exhibits poor solubility in hexane, add approximately 1 mL of benzene with a pipet to the vial to aid in dissolving the sample.
- 11.6.5 Add 1.0 ml of the PAR native spiking solutions (see section 7.11.2) to the LCS/OPR, LCSD (if required), and MS/MSD samples. Record the spike solution identification number and the volume spiked on the sample prep sheet. Initial and date the entry.
- 11.6.6 Proceed to sample extract cleanup in section 11.9.

11.7 Air Sampling Trains

- 11.7.1 For media and sample preparation of air sampling trains refer to Knoxville SOP, KNOX-ID-0012.

11.8 Macro Extract Concentration by Rapid-Vap

- 11.8.1 Preheat the unit to the appropriate temperature for the solvent used in the extraction.
- 11.8.2 Set the operating parameters on the programmer. For example, if there is 300 ml of a methylene chloride extract, the following parameters are used and adjusted as needed:

Temperature	30 °C
Vortex Speed	30%, to be increased at a later time
Nitrogen	7-9 psi
Timer Set	30 minutes

- 11.8.3 Place 600 ml concentrator tubes containing the extract in the Rapid-Vap. Begin concentrating the extract, adjust the vortex speed for the proper rate of concentration.
- 11.8.4 When the extract has been concentrated to less than 20 mL, add approximately 60 mL of hexane. Concentrate the extract to a final volume of approximately 2 mL (that is the volume contained in the reservoir tip of the Rapid-Vap). Shut off the nitrogen flow and turn off the Rapid-Vap or remove the 600 mL concentrator tube to prevent further concentration.
- 11.8.5 Transfer the extract to a 40 mL vial with a 9" disposable pipet, rinsing the sample tube three times with 2 mL of hexane. Reduce the volume in the 40 mL vial using the N-Evap to approximately 2 mL and proceed to extract cleanup in section 11.9. If no cleanups are to be performed continue with the following steps to dry the extract. The sample extracts sometimes contain small amounts of water due to condensation forming on the glassware during the concentration steps. Take care to ensure the water is removed.
- 11.8.6 Prepare a small funnel by placing a small plug of pre-cleaned glass wool at the bottom of the funnel and adding a layer of sodium sulfate on top of the glass wool.
- 11.8.7 Pipet the extract from the Rapi-Vap concentrator tube and through the funnel containing the sodium sulfate into a 40 mL vial.
- 11.8.8 Rinse the concentrator tube 3 times with approximately 3 mL of hexane for each rinse. Rinse the sodium sulfate funnel with an additional 2 mL of hexane. Proceed to micro concentration in section 11.10.
- 11.9 Sample Extract Cleanup
- 11.9.1 Any or all sample cleanups may be employed to remove sample interferences as necessary. The sample batch cleanups must also be performed on the associated quality control samples (method blank, LCS/LCSD). MS/MSD samples must undergo the same cleanup procedures as the associated parent (OS) sample.
- 11.9.2 For 1613B samples that are to be subjected to one or more cleanup steps, add 1.0 mL of the ³⁷Cl-,2,3,7,8-TCDD cleanup standard before any cleanup steps are performed(see section 7.11.6) to each sample extract as well as the method blank and OPR sample extracts.
- 11.9.3 Acid-Base Cleanup
- The acid-base cleanup is employed when sample extracts are colored and/or oily in appearance, or if specified by the client or project manager.
- 11.9.3.1 Bring the extract volume up to ~12 mL with hexane in a 40 mL vial.
- NOTE: If the extracts are from fish tissue, omit sections 11.9.3.2 and 11.9.3.3.
- 11.9.3.2 Wash the extract by adding 10 mL of 20% aqueous potassium hydroxide to the vial and gently shaking for 20 seconds. If an emulsion begins to form,

discontinue shaking. Vent the vial frequently to prevent pressure build up. Let the vial stand for 10 minutes or longer until any emulsion present settles out. Carefully remove the aqueous layer (the bottom layer) with a glass pipet, taking care not to remove any of the solvent layer or remaining emulsion. Repeat the base washing until no color is visible in the base layer (perform a maximum of four base washings).

- 11.9.3.3 Add 10 mL of 5% (w/v) aqueous sodium chloride to the vial and gently shake for 20 seconds. If an emulsion forms, discontinue shaking. Vent the vial frequently to prevent pressure build up. Let the vial stand for 10 minutes or longer until any emulsion present settles out. Carefully remove the aqueous layer (the bottom layer) with a glass pipet, taking care not to remove any of the solvent layer or remaining emulsion.
- 11.9.3.4 Slowly add 15 mL of concentrated sulfuric acid to the vial and shake for 30 seconds. If an emulsion remains from the previous, slowly add concentrated sulfuric acid (drop by drop) to the vial. **CAUTION: HEAT CAN BE GENERATED DURING THIS STEP.** If an emulsion still exists, discontinue shaking. Vent the vial frequently to prevent pressure build up. Let the vial stand for 10 minutes or longer until any emulsion settles out. Carefully remove the aqueous layer (the bottom layer) with a glass pipet, taking care not to remove any of the solvent layer or remaining emulsion. Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings). An option to employ if the acid layer is large or very dark and thick is to remove the Hexane layer (the top layer) and any emulsion to a fresh 40 ml vial. Rinse the vial containing the acid 2 times with 2 ml of Hexane and add these rinsings to the sample vial and then proceed with section 11.9.2.5.
- 11.9.3.5 Add 10 mL 5% (w/v) aqueous sodium chloride to the vial and gently shake for 20 seconds. Vent the vial frequently to prevent pressure build up. Let the vial stand for 10 minutes or longer until any emulsion present settles out. Carefully remove the aqueous layer (the bottom layer) with a glass pipet, taking care not to remove any of the solvent layer or remaining emulsion. Dry the hexane extract by adding 1 to 2 grams of sodium sulfate and swirling the vial.

11.9.3.6 Reduce the extract volume to approximately 2 ml.

11.9.3.7 Proceed to section 11.9.4, silica gel/alumina column cleanup.

11.9.4 Silica Gel/Alumina Column Cleanup

Silica gel/alumina column cleanup is employed when sample extracts are clear or after other cleanup techniques have been employed. If treated drinking water samples are being analyzed, cleanups may not be necessary.

- 11.9.4.1 Prepare a 20 mm diameter column and a 16mm diameter column for each extract by rinsing, in order, with acetone, toluene, methylene chloride and

hexane. Place a large ball of pre-cleaned glass wool in the bottom of each column.

- 11.9.4.2 Mark the level to which the column packings are to be added with a marking pen starting at the top of the glass wool plug and proceeding from bottom to top. The levels for each type column are as follows;

20 mm diameter Silica Gel column:

12 mm – 2g of 3.3% deactivated silica gel

16 mm – 4g of acidic silica gel

12 mm - 2g of 3.3% deactivated silica gel

10 mm – sodium sulfate

16 mm diameter Alumina Column:

40 mm – 6 g of neutral alumina

10 mm – sodium sulfate

- 11.9.4.3 Place the columns to the lab supports in the hood so that the 20 mm silica gel column is above the 16 mm alumina column. Offset the columns slightly so that the packings can be added and the columns rinsed.
- 11.9.4.4 Add the column packing in the order listed above while tapping with a marking pen to column to settle the contents to prevent channeling. When the columns have been completely packed, remove the ink markings with a paper towel moistened with methylene chloride.
- 11.9.4.5 Place a 125 mL glass jar under the lower alumina column to catch the solvent wastes and eluents as they filter through the column.
- 11.9.4.6 Add 20 mL of hexane to each column to rinse the packing. Collect the hexane from the columns in the 125 mL glass jar, the columns must be aligned so that the waste does not drip on the surface of the hood. When the level of solvent in the silica gel column approaches the top of the packing, move the upper column support so that the tips of the upper columns are inserted into the tops of the lower columns and the solvent must drip into the lower columns.
- 11.9.4.7 Just as the level of hexane reaches the top of the packing in the silica gel column transfer the sample extract into the top of the column. Rinse the extract vial 3 times with 1.5 mL of hexane and add each of these rinsings to the silica gel column.
- 11.9.4.8 Just as the solvent level reaches the top of the column packing add 70 mL of hexane (via a solvent bottle top dispenser) into the top of the silica gel column and allow this to drip into and through the alumina column and into the collection jar. When the hexane has completely drained from the silica gel column, remove the column from the support rack and dispose of it in the appropriate waste container.

- 11.9.4.9 Just as the level of hexane reaches the top of the packing in the alumina column using a solvent dispenser add 10ml of 5% methylene chloride/hexane mixture. Immediately after adding the 5% mixture replace the 125 mL glass jar containing the solvent waste with 40ml vial which has been labeled with the sample workorder number. Dispose of the solvent waste in the 125 mL glass jar in the appropriate waste collection container.
- 11.9.4.10 Just as the level of the 5% mixture reaches the top of the packing in the alumina column add 30 mL of 65% methylene chloride/hexane using a solvent dispenser and continue to catch the eluents in the 40 mL vial.
- 11.9.4.11 When the solvent has completely drained from the alumina column, cap the 40 mL vial containing the eluent and dispose of the alumina column in the appropriate waste container.
- 11.9.4.12 If no further cleanup is to be performed, proceed to final extract micro concentration detailed in Section 11.10. Otherwise, reduce the volume of the extract to approximately 2 mL using the nitrogen micro concentration apparatus and proceed to the next cleanup.

11.9.5 Activated Carbon Cleanup

Carbon column cleanups are performed to remove diphenyl ether interferences and when site history indicates they are necessary for removal of other interferences. Carbon column cleanups are also performed when sample extracts cause the acid silica layer of the dual column cleanup to become colored along the entire length of the acid silica. Most solid samples meet these criteria.

- 11.9.5.1 Prepare a disposable carbon column by cutting off the tapered end of a column to achieve a column approximately 20 cm in length. Insert a glass-wool plug of about 1 cm in length approximately 10 cm into the column and pack the column with 5.0 cm of the J.T Baker Carbon/Silica Gel mixture (section 7.7.8). Hold the packing by inserting an additional glass wool plug, again about 1 cm in length, in the other end.
- 11.9.5.2 Pre-elute the column with 10 mL of cyclohexane/methylene chloride (50:50 v/v). Turn the column over and pre-elute in the opposite direction with another 5 mL of cyclohexane/methylene chloride (50:50 v:v).
- 11.9.5.3 When the solvent reaches the glass wool, add the sample extract. Rinse the sample vial 2 times with 2 mL of 50/50 cyclohexane/methylene chloride. Add these rinses to the column. Elute the column with the following sequence of solvents:
 - 11.9.5.3.1 6 mL of cyclohexane/methylene chloride (50:50 v/v).
 - 11.9.5.3.2 5 mL of methylene chloride/methanol/benzene (75:20:5 v/v).
- 11.9.5.4 Allow the 75:20:5 methylene chloride/methanol/benzene to drain completely. Turn the column over and in the direction of reverse flow elute the PCDD/ PCDF fraction with 30 mL toluene into a 40 mL vial.

11.9.5.5 Place vials containing the extract in the nitrogen concentration apparatus and reduce the solvent volume to approximately 0.3 mL.

11.10 Micro Extract Concentration by Nitrogen Blowdown.

11.10.1 When all cleanups have been completed on the sample, using a Hamilton syringe (see section 6.3.3.2) add 20 μ L of the labeled recovery standard spiking solution (see section 7.11.8) to an empty clean 1.1 mL tapered minivial that has been labeled with the sample ID. Mark the level of the recovery standard on the minivial (mark half the level, 10 μ L, if the extracts are from treated drinking waters). Record the volume of recovery standard added on the benchsheet.

11.10.2 Transfer the concentrated extract into the mini-vial. Rinse the 40 mL vial at least twice with a small amount of hexane and add the rinses to the minivial. Put the minivial on the N-EVAP nitrogen blowdown and reduce the volume to the mark on the vial. Put the cap with PTFE-faced septa securely on the vial. Record the final extract volume on the benchsheet.

11.10.3 All items listed on the data review check list must be checked by both the prep analyst who performed the extraction and cleanups and the prep analyst who performed the second level review. An example data review check list is shown in Figure 4.

11.10.4 Transfer the extracts and paperwork to the GC/MS group for analysis.

11.11 Sample Extract Analysis

11.11.1 Analyze the sample extracts under the same instrument operating conditions used to perform the instrument calibrations. Inject 2 μ L into the GC/MS and acquire data until OCDF has eluted from the column.

11.11.2 Record analysis information in the instrument logbook. The following information is required:

Date of analysis

Time of analysis

Instrument data system filename

Analyst

Lab sample identification

Additional information can be recorded in the logbook if necessary.

11.11.3 Generate ion chromatograms for the masses listed in Table 21 that encompass the expected retention windows of the PCDD and PCDF homologous series.

12. Data Analysis and Calculations

12.1 Refer to Figure 4 for an example data review checklists used to perform and document the review of the data. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data.

12.2 Qualitative identification criteria for PCDDs and PCDFs. For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

- 12.2.1 The ion current response for both ions used for quantitative purposes must reach maximum simultaneously (± 2 seconds).
- 12.2.2 The signal-to-noise ratio (S/N) for each GC peak at each exact m/z must be ≥ 2.5 for positive identification of a PCDD/PCDF compound.
- 12.2.3 The ratio of the integrated areas of the two exact m/z's specified in Table 21 must be within the limits specified in Table 22, or alternatively when performing method 1613B, within ± 10 percent of the ratio in the midpoint (CS3) calibration or the calibration verification (VER), whichever is most recent.
- 12.2.4 Method 1613B only - The relative retention time of the peak for a 2,3,7,8-substituted PCDD or PCDF must be within the limits in Table 3.
- 12.2.5 Method 8290, 8290A and 0023A only - For 2,3,7,8-substituted isomers, which have an isotopically labeled internal standard or recovery standard present in the sample extract, the retention time of the two ions used for quantitation purposes must be within -1 to +3 seconds of the isotopically labeled standard.
- 12.2.6 Method 23 and TO-9A only - For 2,3,7,8-substituted isomers, which have an isotopically labeled internal standard or recovery standard present in the sample extract, the retention time of the two ions used for quantitation purposes must be within ± 3 seconds of the isotopically labeled standard.
- 12.2.7 Method 8290, 8290A, 23, 0023A, and TO-9A only - For 2,3,7,8-substituted isomers, which do not have an isotopically labeled internal standard present in the sample extract, the retention time must fall within 0.005 retention time units of the relative retention times measured in the routine calibration.
- 12.2.8 The retention time of peaks representing non-2,3,7,8-substituted PCDDs/PCDFs must be within the retention time windows established in section 10.2.4.
- 12.2.9 No peaks detected in the polychlorinated diphenyl-ether (PCDPE) mass channel in the same retention time region (± 2 sec for method 8290, 8290A & 0023A) as a PCDF peak.

12.3 Quantitation for PCDD's and PCDF's.

- 12.3.1 Calculate the Internal Standard and Cleanup Standard Recoveries (Ris) relative to the Recovery Standard according to the following equation:

$$R_{is} = \frac{A_{is} \times Q_{rs}}{A_{rs} \times RRF_{is} \times Q_{is}} \times 100\%$$

where:

A_{is} = sum of the areas of the quantitation ions of the appropriate internal standard (cleanup standard is single ion)

Ars = sum of the areas of the quantitation ions of the recovery standard
 Qrs = ng of recovery standard added to extract
 Qis = ng of internal standard added to sample
 RRFis = mean relative response factor of internal standard obtained during initial calibration

Note: In some situations, such as high-volume water sampling or air train samples, the extract is split for multiple analyses. In this case, Qrs must be correctly calculated to account for the splitting of extracts before the recovery standard was added.

$$Qrs = \frac{Crs \times Vrs}{S}$$

Where:

Qrs = ng of recovery standard added to extract
 Crs = concentration of recovery standard added to the split portion of the extract
 Vrs = volume of recovery standard added to the split portion of the extract
 S = split ratio of the extract (decimal fraction of the extract used)

- 12.3.2 The split ratio represents the proportion of extract used from splits taken after the addition of internal standards and before the addition of recovery standards. The split ratio is calculated as the product of all split ratios generated between these steps:

$$S = Spis \times Spcs \times Spfc$$

Where:

Spis = the decimal fraction of extract used from split taken once the internal standard has been added and the extraction is performed.
 Spcs = the decimal fraction of extract used from split taken once the cleanup standard (if used) has been added.
 Spfc = the decimal fraction of extract used from split taken once the cleanup fractionation column has been run.

- 12.3.3 When properly applied, isotope dilution techniques produce results that are independent of recovery. The recovery of each internal standard must be within the limits specified in Table 13 for method 1613B, 8290 or 8290A or in Table 15 for method 23, 0023A, or TO-9A. If the recovery of any internal standard is not within the specified limits, calculate the S/N ratio of the internal standard. If the S/N is ≥ 10 and the method minimum levels are met, report the data as is with qualifiers in the report and a discussion in the case narrative. If the S/N is < 10 or the minimum levels are not achieved, re-extract and re-analyze the sample. If the poor internal standard recovery is judged to be a result of sample matrix, a reduced portion of the sample can be re-extracted or additional clean-ups can be employed.

- 12.3.4 Calculate the concentration of the 2,3,7,8 isomers according to the following equation:

$$C_{2,3,7,8 \text{ isomers}} = \frac{Ata \times Qis}{Ais \times RRF \times Ws}$$

Where:

C	=	Concentration of 2,3,7,8 isomers
Ata	=	sum of the areas of the quantitation ions of the target analyte
Ais	=	sum of the areas of the quantitation ions of the appropriate internal standard
Qis	=	ng of internal standard added to sample
RRF	=	mean relative response factor from initial calibration.
Ws	=	amount of sample spiked and extracted (grams or liters)

- 12.3.5 The concentrations of non-2,3,7,8-isomers are calculated using the RRF for the corresponding 2,3,7,8-isomer. If more than one 2,3,7,8-isomer exist for a particular level of chlorination, the average of the individual 2,3,7,8-isomer RRF's is used in the calculation.

$$C_{\text{non 2,3,7,8 isomer}} = \frac{Ata \times Qis}{Ais \times RRF \times Ws}$$

Where:

Ata	=	sum of the areas of the quantitation ions of the non-2,3,7,8 isomer
Ais	=	sum of the areas of the quantitation ions of the appropriate internal standard
Qis	=	ng of internal standard added to sample
RRF	=	mean relative response factor from initial calibration for the corresponding 2,3,7,8 isomer.
Ws	=	amount of sample spiked and extracted (grams or liters)

- 12.3.6 Calculate the total concentration of all isomers within each homologous series of PCDD's and PCDF's by summing the concentrations of the individual PCDD or PCDF 2,3,7,8 and non-2,3,7,8 isomers.

$$C_{\text{Total}} = \sum C_{2,3,7,8 \text{ isomers}} + \sum C_{\text{non 2,3,7,8 isomers}}$$

- 12.3.7 If solid samples are to be reported on a dry weight basis, the laboratory LIMS system performs the following calculation;

$$\text{Concentration (Dry Weight)} = \frac{C}{\% \text{Solids} \div 100}$$

Where:

C	=	Concentration of the target analyte
%Solids	=	The sample percent solids determined by moisture analysis

- 12.3.8 If no peaks are present in the region of the ion chromatogram where the compounds of interest are expected to elute, calculate the estimated detection limit (EDL) for that compound according to the following equation:

$$EDL = \frac{N \times 2.5 \times Qis}{His \times RRFs \times Ws \times Ssl}$$

Where:

N	=	average peak to peak noise of quantitation ion signals in the region of the ion chromatogram where the compound of interest is expected to elute
His	=	peak height of quantitation ions for appropriate internal standard
Qis	=	ng of internal standard added to sample
RRFs	=	mean relative response factor of compound for the shift opening and closing standards
W	=	amount of sample spiked and extracted (grams or liters)
Ssl	=	decimal expression of percent solids (optional, if results are requested to be reported on dry weight basis)

Note: The percent solids calculation is performed by the laboratory LIMS system prior to final reporting.

- 12.3.9 If peaks are present in the region of the ion chromatogram which do not meet the qualitative criteria listed in section 12.2.3, calculate an Estimated Maximum Possible Concentration (EMPC). Two different calculation formulas can be used depending upon specific client requirements.
- 12.3.9.1 When performing methods 8290, 8290A for EPA regulated analyses where the currently promulgated method is required by law (e.g. Trial Burns) and for all other analyses unless the client has specified otherwise, use the equation in section 12.3.4, except that A_{ta} represents the sum of the area under the one peak and of the other peak area calculated using the theoretical chlorine isotope ratio. The peak selected to calculate the theoretical area is the one which gives the lower of the two possible results (i.e. the EMPC is lower than the result calculated from the uncorrected areas).
- 12.3.9.2 When the client has specifically requested, use the equation in section 12.3.4 without correcting the areas. This method gives an EMPC which is always higher than the method above and would be considered the worst case.
- 12.3.10 If peaks are present in the diphenyl ether mass channel at the same retention time as a PCDF peak, the peak cannot be identified as a PCDF. Calculate the concentration of the peak using the equation in section 12.3.4 but report the concentration as an Estimated Maximum Possible Concentration.
- 12.3.11 If the concentration in the final extract of any 2,3,7,8-substituted PCDD/PCDF isomer (except OCDD or OCDF) exceeds the upper method calibration limits, a dilution of the extract or a re-extraction of a smaller portion of the sample must be performed. For the other congeners (including OCDD and OCDF), however, report the measured concentration and indicate that the value exceeds the calibration limit by flagging the results with "E". Dilutions of up to 1/10 can be performed on the extract. If the compounds that exceed the calibration range cannot be brought within the calibration range by a 1/10 dilution, extraction of a smaller aliquot of sample can be performed or the sample can be analyzed by a more appropriate analytical technique such as HRGC/LRMS. Consultation with the client must occur before any re-extraction is performed.

- 12.3.12 Evaluate the ion chromatograms of the PFK lock mass and calibration mass for each MID group. The PFK mass intensity must be consistent throughout the retention time of the target compounds. Negative excursions or variations in the PFK mass intensity indicate the elution of interferences from the GC column that are causing suppression in the ion source of the mass spectrometer. This ion suppression can reduce the instrument sensitivity and quantitative result of any peaks that elute at the same retention time. Either additional extract cleanup or dilutions can reduce ion suppression. The quantitative results must be carefully evaluated when there is evidence of ion suppression present in the PFK mass traces.
- 12.4 The DB-5 (RTX-5) column does not provide for isomer specificity of 2,3,7,8-TCDF using the operating condition required for this method. If a peak is determined to be present at the expected retention time of 2,3,7,8-TCDF and its calculated concentration is above the MinL, the sample extract must be analyzed on the DB-225 (RTX-225) column.
- 12.5 The Minimum Level (MinL) is defined as the level at which the instrument gives acceptable calibration assuming a sample is extracted at the recommended weight or volume and is carried through all normal extraction and analysis procedures. Deviation from the extraction amounts or final volumes listed Table 2 changes the MinL. The MinL is calculated as shown in the following equation:

$$\text{MinL} = \frac{C_{\text{min}} \times V_{\text{fe}}}{W_{\text{s}}}$$

Where:

- C_{min} = the concentration the analyte in the lowest calibration standard
 W_{s} = amount of sample spiked and extracted (grams or liters)
 V_{fe} = the final volume of the extract, corrected for all splits and dilutions

$$V_{\text{fe}} = \frac{V_{\text{del}} \times DF_{\text{pr}}}{Spr \times S}$$

Where:

- V_{del} = the volume of extract delivered to the analysis
 DF_{pr} = the dilution factor for dilutions performed to the final extract
 Spr = the split ratio for any post-recovery standard splits
 S = the split ratio for any post-internal standard and post-cleanup standard splits

- 12.6 The Maximum Level (MaxL) is defined as the concentration or mass of analyte in the sample that corresponds to the highest calibration level in the initial calibration. It is equivalent to the concentration of the highest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed. The MaxL is calculated as shown in the following equation:

$$\text{MaxL} = \frac{C_{\text{max}} \times V_{\text{fe}}}{W_{\text{s}}}$$

Where:

C_{max} = the concentration the analyte in the highest calibration standard V_{fe} and W_s are defined in Section 12.5.

- 12.7 Flag all compound results in the sample that were detected in the method blank with a “B” qualifier.
- 12.8 Flag all compound results in the sample that are below the minimum level with a “J” qualifier.
- 12.9 Flag all compound results in the sample that are above the upper calibration limit with an “E” qualifier.
- 12.10 Flag all compound results in the sample that are “Estimated Maximum Possible Concentrations” with a “Q” qualifier.
- 12.11 Flag compound results in the sample that exhibit chromatographic evidence of co-eluting compounds with a “C” qualifier.
- 12.12 Flag compound results in the sample that are affected by ion suppression with a “S” qualifier.
- 12.13 Data Review
 - 12.13.1 The analyst who performs the initial data calculations must initial and date the front chromatogram of the raw data package to document that they have performed the qualitative and quantitative analysis on the sample data.
 - 12.13.2 A second analyst must verify all qualitative peak identifications. If discrepancies are found, the data must be returned to the analyst who performed the initial peak identification for resolution.
 - 12.13.3 A second analyst must check all hand calculation and data entry into calculation programs, databases, or spreadsheets at a frequency of 100 percent. If discrepancies are found, the data must be returned to the analyst who performed the initial calculation for resolution.
 - 12.13.4 The reviewing analyst must initial and date the front chromatogram of the raw data package to document that they have performed the second level review on the sample data.
 - 12.13.5 All items listed on the data review check list must be checked by both the analyst who performed the initial qualitative and quantitative analysis and the analyst who performed the second level review. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data. An example data review check list is shown in Figure 4.

13. Method Performance

- 13.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. The procedure for determination of the method detection limit is given in the SOP CA-Q-S-006, current revision, based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit. MDL summaries are stored on the local area network.

- 13.2 Initial Demonstration of Capability – Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in Test America Knoxville SOP KNOX-QA-0009.
- 13.3 Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009 current revision for further requirements for performing and documenting initial and on-going demonstrations of capability.

14. Pollution Prevention

- 14.1 All procedures must be conducted in a manner to minimize, as far as practical, the use of solvents, reagents and other chemicals.

15. Waste Management

- 15.1 All waste must be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”
- 15.2 See the current revision of the KNOX-HS-0002 for specific waste handling guidelines.
- 15.3 Waste Streams Produced by the Procedure: The following waste streams are produced when this method is carried out.
- Waste solvents including acetone, toluene, methylene chloride, and hexane from glassware rinsing and sodium sulfate pre-rinsing must be placed in the flammable waste stream, contained in a steel satellite accumulation container type or flammable solvent container.
 - Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials that might or might not be contaminated/hazardous must be placed in the incinerable laboratory waste stream, contained in a poly satellite accumulation container.
 - Extracted PUF filters, XAD-2 resin, paper funnel filters, glass wool, fish/crawfish and soil contaminated with solvents must be placed in the incinerable laboratory waste stream, contained in a poly satellite accumulation container.
 - Contaminated sulfuric acid used during extract cleanup must be placed in the acidic laboratory waste stream, contained in a poly satellite accumulation container or 55 gallon poly drum.
 - Extracted aqueous samples, contaminated with methylene chloride must be placed in the organic water waste stream, contained in a poly satellite accumulation container.
 - Silica gel, alumina, carbon and sodium sulfate, from column clean-ups, contaminated with various solvents and eluates must be placed in the incinerable laboratory waste stream, contained in a poly satellite accumulation container.

16. References

- 16.1 TestAmerica Knoxville Quality Assurance Management (QAM), current revision.

- 16.2 EPA Method 1613: Tetra- Through Octa- Chlorinated Dioxins And Furans by Isotope Dilutions HRGC/HRMS, Revision B, October 1994
- 16.3 USEPA SW-846 "Test Methods for Evaluating Solid Waste" Third Edition, Method 8290, 8290A and 0023A.
- 16.4 USEPA Method 23 – Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans from Municipal Waste Combustors. 40 CFR Part 60 Appendix A.
- 16.5 Method TO-9A: Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition EPA/625/R-96/010b.
- 16.6 TestAmerica Knoxville SOP, KNOX-ID-0012, Method 0023A and Method 0010 Sampling Train Pre-Sampling Preparation and Sample Extraction Procedure (Includes TO-9A Sampling Components).
- 16.7 TestAmerica Knoxville SOP, KNOX-QA-0002, Glassware Cleaning, current revision.
- 16.8 TestAmerica Policy, CA-T-P-002, Selection of Calibration Points, current revision.

17. Miscellaneous

- 17.1 Deviations from Reference Method.
 - 17.1.1 Spiking levels have been reduced to minimize the amount of dioxin contaminated waste generated by this procedure. It has been demonstrated that the performance criteria specified in the method are not affected by this modification.
 - 17.1.2 Method 1613B employs a gravimetric determination of sample size rather than a volumetric determination. This procedure employs a volumetric determination of sample size to allow reporting of sample concentration in the standard units of pg/L (ppq). This modification has no impact on the performance criteria of this method.
 - 17.1.3 The determination of solids content procedure used for aqueous samples is the same as the 1613B procedure used for solid samples rather than the 1613B procedure for aqueous samples. The aqueous sample procedure in 1613B is subject to error if the sample density is not exactly 1.0 g/mL.
 - 17.1.4 The amount of hexane used in the solvent exchange step has been reduced from that specified in the reference methods. The reduction in solvent used is a pollution prevention measure. It has been demonstrated that the performance criteria specified in the method are not affected by this modification.
 - 17.1.5 Method 1613B specifies that the sample bottle is rinsed twice with 5 mL of reagent water after the sample is transferred to the separatory funnel. This procedure specifies that the sample bottle is rinsed three times with methylene chloride after the sample is transferred to the separatory funnel. This modification improves the removal of target compounds from the sample bottle.
 - 17.1.6 The separatory funnel is only rinsed once with methylene chloride after the sample is extracted instead of three times as specified in Method 1613B. The reduction in

solvent used is a pollution prevention measure. It has been demonstrated that the performance criteria specified in the method are not affected by this modification.

- 17.1.7 Toluene volumes and cycle rates for Soxhlet extractors have been optimized for the specific size of glassware used and might not be the same as those specified in the referenced method. It has been demonstrated that the performance criteria specified in the method are not affected by this modification.
- 17.1.8 Soxhlet extracts are not filtered before concentration and solvent exchange. The use of glass wool in the extraction thimbles eliminates the transfer of particles to the extraction solvent. The column cleanup procedures remove any particulate that might not be removed by the glass wool. It has been demonstrated that the performance criteria specified in the methods is not affected by this modification.
- 17.1.9 Particle size determination and reduction as specified in method 1613B is not performed on a routine basis. Silica and sand is not added to the Soxhlet extraction thimble as specified in method 1613B. These procedures are considered to be outside the scope of the laboratories routine extraction procedures and are only performed on a client specific or project specific basis. These procedures, if required, must be specified and documented in the appropriate QAPPs.
- 17.1.10 Benzene is used to aid in dissolving the samples and/or extracts in hexane. It has been demonstrated that the performance criteria specified in the methods is not affected by this modification.
- 17.1.11 The absolute retention time requirements in Method 1613 section 15.4.1.1 is not required in this procedure. The routine maintenance required of GC columns when analyzing samples from hazardous waste sites makes this requirement virtually impossible to meet in a commercial laboratory environment. This requirement provides no additional quality assurance purpose beyond those already provided by the use of labeled internal standards and required relative retention time limits.
- 17.1.12 This procedure provides for additional calculation and reporting of sample specific detection limits and estimated maximum possible concentrations not required by Method 1613. These reporting conventions are similar to those required by EPA SW-846 Method 8290 and expected by data users familiar with EPA Office of Solid Waste program requirements.
- 17.1.13 The acid-base cleanup procedure is carried out in a VOA vial instead of a separatory funnel. Disposable glassware is used to decrease the risk of cross contamination. The volumes of the washes used have been adjusted for use in the VOA vials. It has been demonstrated that the performance criteria specified in the methods is not affected by this modification.
- 17.1.14 The silica gel/alumina column cleanup used in this procedure has been optimized relative to amount and order of packings and can vary from the various columns and packings specified in the referenced methods. The solvent volumes and mixtures have been optimized based on evaluation of the elution of native and labeled standards. The silica gel and alumina are heated in an oven at 130°C instead of

110°C. It has been demonstrated that the performance criteria specified in the methods is not affected by this modification.

- 17.1.15 The carbon column used in this procedure is based on the column specified in method 8280. Silica gel is used as the carbon column support instead of Celite 545[®] as specified in methods 8290 and 1613B. It has been determined that silica gel is less likely to contain contaminants and interferences which are not removed by the precleaning procedures than Celite 545[®], yet performs similarly. The solvents and elution schemes used are as specified in method 8280 rather than 8290 and 1613B. It has been demonstrated that the performance criteria specified in the methods is not affected by this modification.
- 17.1.16 Methods 8290/8290A do not require dilution and reanalysis of samples for which OCDD exceeds the calibration range. Although this allowance is not made by method 1613B, this procedure does not require dilution for OCDD on samples analyzed by that method.
- 17.1.17 The calibration standards specified in method 23 are used for method 0023A and TO-9A.
- 17.1.18 Extracts are stored at room temperature rather than at <10 °C. The reference method requires that standards be stored at room temperature. Recovery studies performed by Cambridge Isotopes Laboratories (CIL) indicate freezing or refrigeration of standards causes problems with precipitation and irreversible adsorption to the inside surface of the vial. CIL recommends the storage of standards and extracts at room temperature as long as they are protected from exposure to UV and evaporative losses.
- 17.2 Summary of modifications to SOP
- 17.3 Summary of revisions to SOP for revision 8
 - 17.3.1 Changed SOP to reflect EPA Method SW-846 8290A.
- 17.4 Summary of revisions to SOP for revision 7
 - 17.4.1 Changed: "Analyze 2µL of the Initial Calibration Verification (ICV) Standard in section 7.9.2 after the completion of the initial calibration prior to sample analysis.
 - 17.4.2 Added the preparation of sodium sulfate and its vendor in sections 7.2.7.
 - 17.4.3 Added the amount of potassium hydroxide and DI water in 7.5.2
 - 17.4.4 Added the amounts of sulfuric acid, DI water, silica gel, and methylene chloride in sections 7.6.7, 7.6.7, 7.6.11, 7.7.1
 - 17.4.5 Added vendor for activated carbon and activation process in 7.7.2 and 7.7.3
 - 17.4.6 Added PAR native solution in 7.9.4
 - 17.4.7 Added standard solution information in 7.10.2, 7.10.3, 7.10.5 and 7.11.2, 7.11.8
 - 17.4.8 Added the grade and preparation of silica gel in 7.6.6

- 17.4.9 Added screening process in 11.2
- 17.4.10 Edited aqueous and solid extraction process.
- 17.5 Summary of revisions to SOP for revision 6.
 - 17.5.1 Added an exception to 1613B allowing OCDD over calibration range.
 - 17.5.2 Added RLs for biological tissues in Table 2.
 - 17.5.3 Added health and safety information to section 5.10 and waste handling information to section 15.2.
 - 17.5.4 Changed the preparation of sodium sulfate in sections 7.2.7, 7.3.8, 7.5.6, 7.6.1.
 - 17.5.5 Changed the oven temperature for alumina and silica gel in section 7.6.6 and 7.6.9.
- 17.6 Summary of revisions to SOP for revision 5.
 - 17.6.1 Added information to sections 3.37, 8.1, 12.1, 13.1, Appendix I.
- 17.7 Summary of revisions to SOP for revision 4.
 - 17.7.1 Corrected the %D for 13C12-1,2,3,6,7,8-HxCDD and -HxCDF for 23 and TO-9A. Updated sections 5 and 15 to meet corporate EH&S requirements.
- 17.8 Summary of modifications to SOP for revision 3.
 - 17.8.1 Incorporated all PCDD/PCDF analysis methods including 8290, 23, 0023A, and TO-9A into this method.
- 17.8.2 Revised all Tables to reflect requirements for each analysis method.
- 17.8.3 Removed instruction to follow carbon cleanup with silica gel/alumina cleanup.
- 17.8.4 Modified the solids determination procedure in section 11.4.9.1 from that specified by method 1613B.
- 17.9 List of tables and figures referenced in the body of the SOP.
 - 17.9.1 Table 1 – Polychlorinated Dibenzodioxins and Furans Determined by Isotope Dilution and Internal Standard High Resolution Gas Chromatography /High Resolution Mass Spectrometry (HRGC/HRMS)
 - 17.9.2 Table 2 – Methods – All, Minimum Levels by Matrix
 - 17.9.3 Table 3 – Methods – 1613B and 8290, Retention Time References, Quantitation References, and Relative Retention Times
 - 17.9.4 Table 4 – Methods – 23, 0023A, and TO-9A, Retention Time References and Quantitation References.
 - 17.9.5 Table 5 – Methods – 1613B and 8290, Initial Calibration Standard Concentrations and Acceptance Criteria.

- 17.9.6 Table 6 – Methods – 23, 0023A, and TO-9A, Initial Calibration Standard Concentrations and Acceptance Criteria.
- 17.9.7 Table 7 – Methods – 1613B and 8290, Daily Verification Standard (VER) Concentrations and Acceptance Criteria.
- 17.9.8 Table 8 – Methods – 23, 0023A, and TO-9A, Daily Verification Standard (VER) Concentrations and Acceptance Criteria.
- 17.9.9 Table 9 – Method – 1613B, Initial Precision and Recovery (IPR) Acceptance Criteria.
- 17.9.10 Table 10 – Methods – 8290, 23, 0023A, and TO-9A, Initial Precision and Recovery (IPR) Acceptance Criteria.
- 17.9.11 Table 11 – Laboratory Control Sample (LCS/OPR) Spiking Solution Component Concentrations and Acceptance Limits.
- 17.9.12 Table 12 – Method – 8290. Matrix Spike and Matrix Spike Duplicate Sample (MS/MSD) Spiking Solution Component Concentrations and Acceptance Limits.
- 17.9.13 Table 13- Methods – 1613B and 8290, Internal Standard Spiking Solution Component Concentrations and Acceptance Limits.
- 17.9.14 Table 14 – Method – 1613B, Cleanup Standard Spiking Solution Component Concentrations and Acceptance Limits.
- 17.9.15 Table 15 – Methods – 23, 0023A, and TO-9A, Internal Standard Spiking Solution Component Concentrations and Acceptance Limits.
- 17.9.16 Table 16 – Methods – 23, 0023A, and TO-9A, Surrogate Standard Spiking Solution Component Concentrations and Acceptance Limits.
- 17.9.17 Table 17 – Methods – All, Recovery Standard Spiking Solution Component Concentrations.
- 17.9.18 Table 18 – Rtx-5/DB-5 Column Window Defining Standard Mixture Components. – Rtx-5 (DB-5) Column Performance Standard Mixture Components.
- 17.9.19 Table 21 – DB-225 (Rtx-225) Column Performance Standard Mixture Components.
- 17.9.20 Table 21 – Ions Monitored for HRGC/HRMS Analysis of PCDDs and PCDFs.
- 17.9.21 Table 22 – Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs.
- 17.9.22 Figure 1 – Recommended GC Operating Conditions.
- 17.9.23 Figure 2 – Recommended MID Descriptors.
- 17.9.24 Figure 3 – Example Sample Prep Benchsheet
- 17.9.25 Figure 4 – Example Data Review Checklist.
- 17.9.26 Figure 5 – Aqueous sample Extraction Flowchart

17.9.27 Figure 6 – Solid Sample Extraction Flowchart

17.9.28 Figure 7 – Sample Cleanup Flowchart

17.9.29 Figure 8 – Analysis of PCDD's and PCDF's by HRGC/HRMS Flowchart

17.9.30 Appendix I Lipid Determination

17.9.31 Attachment I LRMS dioxin Screen Strategy

17.9.32 Attachmend II Example of Determination of Sediment Sample Extraction Amounts

17.9.33 Attachment III Example Gravimetric Lipids Data Worksheet

History of Revisions

HISTORY OF REVISION PAGE

REV NO.	DATE	PAGES AFFECTED	REASON FOR REVISION
0	02/26/97	All	Initial version of the SOP
1	08/31/99	All	Procedure review.
2	01/28/02	All	Procedure review.
3	04/26/03	All	Procedure review
4	11/12/03	56 of 83	Corrected the %D for $^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD and – HxCDF for 23 and TO-9A. Updated sections 5 and 15 to meet corporate EH&S requirements.
5	6/18/04	All	Added information to sections 3.37, 8.1, 12.1, 13.1, Appendix I.
6	9/27/05	All	Added an exception to 1613B allowing OCDD over calibration range. Added health and safety information to section 5.10 and waste handling information to section 15.2. Updated reagent preparation information for sodium sulfate, alumina and silica gel. Added RLs for biological tissues in Table 2.
7	2/17/07	All	Added dioxin screening and moisture sections in order to determine the appropriate sample dry weight for extraction. Edited reagent and standard preparation sections.
8	3/11/09	All	Updated SOP to reflect EPA Method 8290A requirements.

Table 1

**Polychlorinated Dibenzodioxins and Furans Determined by Isotope Dilution and Internal Standard
High Resolution Gas Chromatography /High Resolution Mass Spectrometry (HRGC/HRMS)**

PCDD's/PCDF's ¹ Isomer/Congener	CAS Registry	Labeled Analog	CAS Registry
2,3,7,8-TCDD	1746-01-6	¹³ C ₁₂ -2,3,7,8-TCDD ³⁷ Cl ₄ -2,3,7,8-TCDD	76523-40-5 85508-50-5
Total TCDD	41903-57-5		
2,3,7,8-TCDF	51207-31-9	¹³ C ₁₂ -2,3,7,8-TCDF	89059-46-1
Total TCDF	55722-27-5		
1,2,3,7,8-PeCDD	40321-76-4	¹³ C ₁₂ -1,2,3,7,8-PeCDD	109719-79-1
Total PeCDD	36088-22-9		
1,2,3,7,8-PeCDF	57117-41-6	¹³ C ₁₂ -1,2,3,7,8-PeCDF	109719-77-9
2,3,4,7,8-PeCDF	57117-31-4	¹³ C ₁₂ -2,3,4,7,8-PeCDF	116843-02-8
Total PeCDF	30402-15-4		
1,2,3,4,7,8-HxCDD	39227-28-6	¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	109719-80-4
1,2,3,6,7,8-HxCDD	57653-85-7	¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	109719-81-5
1,2,3,7,8,9-HxCDD	19408-74-3	¹³ C ₁₂ -1,2,3,7,8,9-HxCDD	109719-82-6
Total HxCDD	34465-46-8		
1,2,3,4,7,8-HxCDF	70648-26-9	¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	114423-98-2
1,2,3,6,7,8-HxCDF	57117-44-9	¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	116843-03-9
2,3,4,6,7,8-HxCDF	60851-34-5	¹³ C ₁₂ -2,3,4,6,7,8-HxCDF	116843-05-1
1,2,3,7,8,9-HxCDF	72918-21-9	¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	116843-04-0
Total HxCDF	55684-94-1		
1,2,3,4,6,7,8-HpCDD	35822-46-9	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	109719-83-7
Total HpCDD	37871-00-4		
1,2,3,4,6,7,8-HpCDF	67562-39-4	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	109719-84-8
1,2,3,4,7,8,9-HpCDF	55673-89-7	¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	109719-94-0
Total HpCDF	38998-75-3		
OCDD	3268-87-9	¹³ C ₁₂ -OCDD	114423-97-1
OCDF	39001-02-0	none	

Notes:

1. Polychlorinated dioxins and furans

TCDD = Tetrachlorodibenzo-p-dioxin
 PeCDD = Pentachlorodibenzo-p-dioxin
 HxCDD = Hexachlorodibenzo-p-dioxin
 HpCDD = Heptachlorodibenzo-p-dioxin
 OCDD = Octachlorodibenzo-p-dioxin

TCDF = Tetrachlorodibenzofuran
 PeCDF = Pentachlorodibenzofuran
 HxCDF = Hexachlorodibenzofuran
 HpCDF = Heptachlorodibenzofuran
 OCDF = Octachlorodibenzofuran

Table 2
Methods – All
Minimum Levels by Matrix

Analyte	Extract (ng/mL) ¹	Water (pg/L) ²	Solids (pg/g) ³	Biological Tissue (pg/g) ³	Waste (pg/g) ⁴	Air/Wipe (pg) ⁵
2,3,7,8-TCDD	0.5	10	1	1	10	10
2,3,7,8-TCDF	0.5	10	1	1	10	10
1,2,3,7,8-PeCDD	2.5	50	5	5	50	50
1,2,3,7,8-PeCDF	2.5	50	5	5	50	50
2,3,4,7,8-PeCDF	2.5	50	5	5	50	50
1,2,3,4,7,8-HxCDD	2.5	50	5	5	50	50
1,2,3,6,7,8-HxCDD	2.5	50	5	5	50	50
1,2,3,7,8,9-HxCDD	2.5	50	5	5	50	50
1,2,3,4,7,8-HxCDF	2.5	50	5	5	50	50
1,2,3,6,7,8-HxCDF	2.5	50	5	5	50	50
2,3,4,6,7,8-HxCDF	2.5	50	5	5	50	50
1,2,3,7,8,9-HxCDF	2.5	50	5	5	50	50
1,2,3,4,6,7,8-HpCDD	2.5	50	5	5	50	50
1,2,3,4,6,7,8-HpCDF	2.5	50	5	5	50	50
1,2,3,4,7,8,9-HpCDF	2.5	50	5	5	50	50
OCDD	5.0	100	10	10	100	100
OCDF	5.0	100	10	10	100	100

Notes:

- 1 Concentration in the extract assuming a 20 µL volume.
- 2 Based on a sample volume of 1.0 L.
- 3 Based on a sample volume of 10.0 g.
- 4 Based on a sample volume of 1.0g.
- 5 Based on extraction of the entire sample.

Table 3
Methods – 1613B and 8290/8290A
Retention Time References, Quantitation References, and Relative Retention Times

Analyte	Retention Time and Quantitation Reference	Relative Retention Time
<i>Compounds using $^{13}\text{C}_{12}$-1,2,3,4-TCDD as the recovery standard</i>		
2,3,7,8-TCDD	$^{13}\text{C}_{12}$ -2,3,7,8-TCDD	0.999-1.002
2,3,7,8-TCDF	$^{13}\text{C}_{12}$ -2,3,7,8-TCDF	0.999-1.003
1,2,3,7,8-PeCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD	0.999-1.002
1,2,3,7,8-PeCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF	0.999-1.002
2,3,4,7,8-PeCDF	$^{13}\text{C}_{12}$ -2,3,4,7,8-PeCDF	0.999-1.002
$^{13}\text{C}_{12}$ -2,3,7,8-TCDD	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD	0.976-1.043
$^{37}\text{Cl}_4$ -2,3,7,8-TCDD	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD	0.989-1.052
$^{13}\text{C}_{12}$ -2,3,7,8-TCDF	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD	0.923-1.103
$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD	1.000-1.567
$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD	1.000-1.425
$^{13}\text{C}_{12}$ -2,3,4,7,8-PeCDF	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD	1.011-1.526
<i>Compounds using $^{13}\text{C}_{12}$-1,2,3,7,8,9-HxCDD as the recovery standard</i>		
1,2,3,4,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDD	0.999-1.001
1,2,3,6,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD	0.998-1.004
1,2,3,7,8,9-HxCDD	-- ¹	1.000-1.019
1,2,3,4,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF	0.999-1.001
1,2,3,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF	0.997-1.005
2,3,4,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -2,3,4,6,7,8-HxCDF	0.999-1.001
1,2,3,7,8,9-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDF	0.999-1.001
1,2,3,4,6,7,8-HpCDD	$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDD	0.999-1.001
1,2,3,4,6,7,8-HpCDF	$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF	0.999-1.001
1,2,3,4,7,8,9-HpCDF	$^{13}\text{C}_{12}$ -1,2,3,4,7,8,9-HpCDF	0.999-1.001
OCDD	$^{13}\text{C}_{12}$ -OCDD	0.999-1.001
OCDF	$^{13}\text{C}_{12}$ -OCDD	0.999-1.008
$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	0.977-1.000
$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	0.981-1.003
$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	0.944-0.970
$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	0.949-0.975
$^{13}\text{C}_{12}$ -2,3,4,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	0.959-1.021
$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	0.977-1.047
$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	1.086-1.110
$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	1.043-1.085
$^{13}\text{C}_{12}$ -1,2,3,4,7,8,9-HpCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	1.057-1.151
$^{13}\text{C}_{12}$ -OCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD	1.032-1.311

Notes:

- 1 The retention time reference for 1,2,3,7,8,9-HxCDD is $^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD. 1,2,3,7,8,9-HxCDD is quantified using the averaged responses for $^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDD and $^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD.

Table 4
Methods – 23, 0023A, and TO-9A
Retention Time References and Quantitation References

Analyte	Retention Time and Quantitation Reference
<i>Compounds using $^{13}\text{C}_{12}$-1,2,3,4-TCDD as the recovery standard</i>	
2,3,7,8-TCDD	$^{13}\text{C}_{12}$ -2,3,7,8-TCDD
2,3,7,8-TCDF	$^{13}\text{C}_{12}$ -2,3,7,8-TCDF
1,2,3,7,8-PeCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD
1,2,3,7,8-PeCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF
$^{13}\text{C}_{12}$ -2,3,7,8-TCDD	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD
$^{37}\text{Cl}_4$ -2,3,7,8-TCDD	$^{13}\text{C}_{12}$ -2,3,7,8-TCDD
$^{13}\text{C}_{12}$ -2,3,7,8-TCDF	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD
$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD
$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF	$^{13}\text{C}_{12}$ -1,2,3,4-TCDD
$^{13}\text{C}_{12}$ -2,3,4,7,8-PeCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF
<i>Compounds using $^{13}\text{C}_{12}$-1,2,3,7,8,9-HxCDD as the recovery standard</i>	
1,2,3,4,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD
1,2,3,4,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF
1,2,3,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF
1,2,3,4,6,7,8-HpCDD	$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDD
1,2,3,4,6,7,8-HpCDF	$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF	$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF
OCDD	$^{13}\text{C}_{12}$ -OCDD
OCDF	$^{13}\text{C}_{12}$ -OCDD
$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD
$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD
$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF
$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD
$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD
$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD
$^{13}\text{C}_{12}$ -1,2,3,4,7,8,9-HpCDF	$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF
$^{13}\text{C}_{12}$ -OCDD	$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD

Table 5
Methods – 1613B and 8290/8290A
Initial Calibration Standard Concentrations and Acceptance Criteria

	CS1	CS2	CS3	CS4	CS5	1613B	8290	8290A
Analyte	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	%RSD	%RSD	%RSD
<u>Native PCDD's and PCDF's</u>								
2,3,7,8-TCDD	0.5	2.0	10	40	200	±20	±20	±20
2,3,7,8-TCDF	0.5	2.0	10	40	200	±20	±20	±20
1,2,3,7,8-PeCDD	2.5	10	50	200	1000	±20	±20	±20
1,2,3,7,8-PeCDF	2.5	10	50	200	1000	±20	±20	±20
2,3,4,7,8-PeCDF	2.5	10	50	200	1000	±20	±20	±20
1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000	±20	±20	±20
1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000	±20	±20	±20
1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000	±35	±20	±20
1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000	±20	±20	±20
1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000	±20	±20	±20
2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000	±20	±20	±20
1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000	±20	±20	±20
1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000	±20	±20	±20
1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000	±20	±20	±20
1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000	±20	±20	±20
OCDD	5.0	20	100	400	2000	±20	±20	±20
OCDF	5.0	20	100	400	2000	±35	±20	±20
<u>Labeled Internal Standards</u>								
¹³ C ₁₂ -2,3,7,8-TCDD	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -2,3,7,8-TCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,7,8-PeCDD	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,7,8-PeCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -2,3,4,7,8-PeCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -2,3,4,6,7,8-HxCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	100	100	100	100	100	±35	±30	±20
¹³ C ₁₂ -OCDD	200	200	200	200	200	±35	±30	±20
<u>Labeled Cleanup Standard</u>								
³⁷ Cl ₄ -2,3,7,8-TCDD	0.5	2.0	10	40	200	±35	-	-
<u>Labeled Recovery Standard</u>								
¹³ C ₁₂ -1,2,3,4-TCDD	100	100	100	100	100	-	-	-
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD	100	100	100	100	100	-	-	-

Table 6

Methods – 23, 0023A, and TO-9A
Initial Calibration Standard Concentrations and Acceptance Criteria

	CS1	CS2	CS3	CS4	CS5	23 / TO-9A	0023A
Analyte	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	%RSD	%RSD
Native PCDD's and PCDF's							
2,3,7,8-TCDD	0.5	1.0	5	50	100	±25	±20
2,3,7,8-TCDF	0.5	1.0	5	50	100	±25	±20
1,2,3,7,8-PeCDD	2.5	5	25	250	500	±25	±20
1,2,3,7,8-PeCDF	2.5	5	25	250	500	±25	±20
2,3,4,7,8-PeCDF	2.5	5	25	250	500	±25	±20
1,2,3,4,7,8-HxCDD	2.5	5	25	250	500	±25	±20
1,2,3,6,7,8-HxCDD	2.5	5	25	250	500	±25	±20
1,2,3,7,8,9-HxCDD	2.5	5	25	250	500	±25	±20
1,2,3,4,7,8-HxCDF	2.5	5	25	250	500	±25	±20
1,2,3,6,7,8-HxCDF	2.5	5	25	250	500	±25	±20
2,3,4,6,7,8-HxCDF	2.5	5	25	250	500	±25	±20
1,2,3,7,8,9-HxCDF	2.5	5	25	250	500	±25	±20
1,2,3,4,6,7,8-HpCDD	2.5	5	25	250	500	±25	±20
1,2,3,4,6,7,8-HpCDF	2.5	5	25	250	500	±25	±20
1,2,3,4,7,8,9-HpCDF	2.5	5	25	250	500	±25	±20
OCDD	5.0	10	50	500	1000	±25	±20
OCDF	5.0	10	50	500	1000	±30	±20
Labeled Internal Standards							
¹³ C ₁₂ -2,3,7,8-TCDD	100	100	100	100	100	±25	±30
¹³ C ₁₂ -2,3,7,8-TCDF	100	100	100	100	100	±30	±30
¹³ C ₁₂ -1,2,3,7,8-PeCDD	100	100	100	100	100	±30	±30
¹³ C ₁₂ -1,2,3,7,8-PeCDF	100	100	100	100	100	±30	±30
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	100	100	100	100	100	±25	±30
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	100	100	100	100	100	±30	±30
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	100	100	100	100	100	±30	±30
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	100	100	100	100	100	±30	±30
¹³ C ₁₂ -OCDD	200	200	200	200	200	±30	±30
Surrogate Standards							
³⁷ Cl ₄ -2,3,7,8-TCDD	0.5	1.0	5	50	100	±25	±30
¹³ C ₁₂ -2,3,4,7,8-PeCDF	2.5	5	25	250	500	±25	±30
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	2.5	5	25	250	500	±25	±30
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	2.5	5	25	250	500	±25	±30
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	2.5	5	25	250	500	±25	±30
Labeled Recovery Standard							
¹³ C ₁₂ -1,2,3,4-TCDD	100	100	100	100	100	-	-
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD	100	100	100	100	100	-	-

Table 7
Methods – 1613B and 8290/8290A
Daily Verification Standard (VER) Concentrations and Acceptance Criteria

Analyte	VER (ng/mL)	1613B			8290/8290A	
		All Isomers (ng/mL)	Tetra only (ng/mL)		Shift Open %D	Shift Close ¹ %D
<u>Native PCDD's and PCDF's</u>						
2,3,7,8-TCDD	10	7.8-12.9	8.2-12.3		±20	±25
2,3,7,8-TCDF	10	8.4-12.0	8.6-11.6		±20	±25
1,2,3,7,8-PeCDD	50	39-65	-		±20	±25
1,2,3,7,8-PeCDF	50	41-60	-		±20	±25
2,3,4,7,8-PeCDF	50	41-61	-		±20	±25
1,2,3,4,7,8-HxCDD	50	39-64	-		±20	±25
1,2,3,6,7,8-HxCDD	50	39-64	-		±20	±25
1,2,3,7,8,9-HxCDD	50	41-61	-		±20	±25
1,2,3,4,7,8-HxCDF	50	45-56	-		±20	±25
1,2,3,6,7,8-HxCDF	50	44-57	-		±20	±25
2,3,4,6,7,8-HxCDF	50	44-57	-		±20	±25
1,2,3,7,8,9-HxCDF	50	45-56	-		±20	±25
1,2,3,4,6,7,8-HpCDD	50	43-58	-		±20	±25
1,2,3,4,6,7,8-HpCDF	50	45-55	-		±20	±25
1,2,3,4,7,8,9-HpCDF	50	43-58	-		±20	±25
OCDD	100	79-126	-		±20	±25
OCDF	100	63-159	-		±20	±25
<u>Labeled Internal Standards</u>						
¹³ C ₁₂ -2,3,7,8-TCDD	100	82-121	85-117		±30	±35
¹³ C ₁₂ -2,3,7,8-TCDF	100	71-140	76-131		±30	±35
¹³ C ₁₂ -1,2,3,7,8-PeCDD	100	62-160	-		±30	±35
¹³ C ₁₂ -1,2,3,7,8-PeCDF	100	76-130	-		±30	±35
¹³ C ₁₂ -2,3,4,7,8-PeCDF	100	77-130	-		±30	±35
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	100	85-117	-		±30	±35
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	100	85-118	-		±30	±35
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	100	76-131	-		±30	±35
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	100	70-143	-		±30	±35
¹³ C ₁₂ -2,3,4,6,7,8-HxCDF	100	73-137	-		±30	±35
¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	100	74-135	-		±30	±35
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	100	72-138	-		±30	±35
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	100	78-129	-		±30	±35
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	100	77-129	-		±30	±35
¹³ C ₁₂ -OCDD	200	96-415	-		±30	±35
<u>Labeled Cleanup Standard</u>						
³⁷ Cl ₄ -2,3,7,8-TCDD	10	7.9-12.7	8.3-12.1		-	-
<u>Labeled Recovery Standard</u>						
¹³ C ₁₂ -1,2,3,4-TCDD	100	-	-		-	-
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD	100	-	-		-	-

Notes:

- 1 If the closing standard %D exceeds the opening %D criteria, the average of the Opening and Closing RF is used instead of the Initial Calibration RF to calculate sample concentrations.

Table 8
Methods – 23, 0023A, and TO-9A
Daily Verification Standard (VER) Concentrations and Acceptance Criteria

Analyte	VER (ng/mL)	23 and TO-9A %D	0023A	
			Shift Open %D	Shift Close ¹ %D
Native PCDD's and PCDF's				
2,3,7,8-TCDD	5	±25	±20	±25
2,3,7,8-TCDF	5	±25	±20	±25
1,2,3,7,8-PeCDD	25	±25	±20	±25
1,2,3,7,8-PeCDF	25	±25	±20	±25
2,3,4,7,8-PeCDF	25	±25	±20	±25
1,2,3,4,7,8-HxCDD	25	±25	±20	±25
1,2,3,6,7,8-HxCDD	25	±25	±20	±25
1,2,3,7,8,9-HxCDD	25	±25	±20	±25
1,2,3,4,7,8-HxCDF	25	±25	±20	±25
1,2,3,6,7,8-HxCDF	25	±25	±20	±25
2,3,4,6,7,8-HxCDF	25	±25	±20	±25
1,2,3,7,8,9-HxCDF	25	±25	±20	±25
1,2,3,4,6,7,8-HpCDD	25	±25	±20	±25
1,2,3,4,6,7,8-HpCDF	25	±25	±20	±25
1,2,3,4,7,8,9-HpCDF	25	±25	±20	±25
OCDD	50	±25	±20	±25
OCDF	50	±30	±20	±25
Labeled Internal Standards				
¹³ C ₁₂ -2,3,7,8-TCDD	100	±25	±30	±35
¹³ C ₁₂ -2,3,7,8-TCDF	100	±30	±30	±35
¹³ C ₁₂ -1,2,3,7,8-PeCDD	100	±30	±30	±35
¹³ C ₁₂ -1,2,3,7,8-PeCDF	100	±30	±30	±35
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	100	±25	±30	±35
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	100	±30	±30	±35
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	100	±30	±30	±35
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	100	±30	±30	±35
¹³ C ₁₂ -OCDD	200	±30	±30	±35
Surrogate Standards				
³⁷ Cl ₄ -2,3,7,8-TCDD	5	±25	±30	±35
¹³ C ₁₂ -2,3,4,7,8-PeCDF	25	±25	±30	±35
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	25	±25	±30	±35
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	25	±25	±30	±35
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	25	±25	±30	±35
Labeled Recovery Standard				
¹³ C ₁₂ -1,2,3,4-TCDD	100	-	-	
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD	100	-	-	

Notes:

- 1 If the closing standard %D exceeds the opening %D criteria, the average of the Opening and Closing RF is used instead of the Initial Calibration RF to calculate sample concentrations.

Table 9
Method – 1613B
Initial Precision and Recovery (IPR) Acceptance Criteria

Initial Precision and Recovery (I/R) Acceptance Criteria						
	Test	1613B			1613B Tetra Only	
	Conc.	s ²	X ³		s ²	X ³
Analyte	(ng/mL) ¹	(ng/mL) ¹	(ng/ml) ¹		(ng/mL) ¹	(ng/ml) ¹
<u>Native PCDD's and PCDF's</u>						
2,3,7,8-TCDD	10	2.8	8.3-12.9		2.7	8.7-12.4
2,3,7,8-TCDF	10	2.0	8.7-13.7		2.0	9.1-13.1
1,2,3,7,8-PeCDD	50	7.5	38-66		-	-
1,2,3,7,8-PeCDF	50	7.5	43-62		-	-
2,3,4,7,8-PeCDF	50	8.6	36-75		-	-
1,2,3,4,7,8-HxCDD	50	9.4	39-76		-	-
1,2,3,6,7,8-HxCDD	50	7.7	42-62		-	-
1,2,3,7,8,9-HxCDD	50	11.1	37-71		-	-
1,2,3,4,7,8-HxCDF	50	8.7	41-59		-	-
1,2,3,6,7,8-HxCDF	50	6.7	46-60		-	-
2,3,4,6,7,8-HxCDF	50	7.4	37-74		-	-
1,2,3,7,8,9-HxCDF	50	6.4	42-61		-	-
1,2,3,4,6,7,8-HpCDD	50	7.7	38-65		-	-
1,2,3,4,6,7,8-HpCDF	50	6.3	45-56		-	-
1,2,3,4,7,8,9-HpCDF	50	8.1	43-63		-	-
OCDD	100	19	89-127		-	-
OCDF	100	27	74-146		-	-
<u>Labeled Internal Standards</u>						
¹³ C ₁₂ -2,3,7,8-TCDD	50	18.5	14-67		17.5	16-57.5
¹³ C ₁₂ -2,3,7,8-TCDF	50	17.5	15.5-56.5		17	17.5-49.5
¹³ C ₁₂ -1,2,3,7,8-PeCDD	50	19.5	13.5-92		-	-
¹³ C ₁₂ -1,2,3,7,8-PeCDF	50	17.0	13.5-78		-	-
¹³ C ₁₂ -2,3,4,7,8-PeCDF	50	19.0	8-139.5		-	-
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	50	20.5	14.5-73.5		-	-
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	50	19.0	17-61		-	-
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	50	21.5	13.5-76		-	-
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	50	17.5	15-61		-	-
¹³ C ₁₂ -2,3,4,6,7,8-HxCDF	50	18.5	14.5-68		-	-
¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	50	20.0	12-78.5		-	-
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	50	17.5	17-64.5		-	-
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	50	20.5	16-55		-	-
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	50	20.0	14-70.5		-	-
¹³ C ₁₂ -OCDD	100	47.5	20.5-138		-	-
<u>Labeled Cleanup Standard</u>						
³⁷ Cl ₄ -2,3,7,8-TCDD	10	3.6	3.9-15.4		3.4	4.5-13.4

Notes:

- 1 All specifications are given as concentration in the final extract, assuming a 20-μL volume.
- 2 s = standard deviation of the concentration
- 3 X = average concentration. The acceptance range for average recovery can be normalized (shifted to center on 100% recovery) to compensate for the bias in the collaborative study used to develop the acceptance criteria.

Table 10
Methods – 8290, 23, 0023A, and TO-9A
Initial Precision and Recovery (IPR) Acceptance Criteria

Analyte	Test Conc (ng/mL) ¹	s ² (%Rec)	X ³ (%Rec)
<u>Native PCDD's and PCDF's</u>			
2,3,7,8-TCDD	10	15 ⁴	70-130 ⁴
2,3,7,8-TCDF	10	15 ⁴	70-130 ⁴
1,2,3,7,8-PeCDD	50	15 ⁴	70-130 ⁴
1,2,3,7,8-PeCDF	50	15 ⁴	70-130 ⁴
2,3,4,7,8-PeCDF	50	15 ⁴	70-130 ⁴
1,2,3,4,7,8-HxCDD	50	15 ⁴	70-130 ⁴
1,2,3,6,7,8-HxCDD	50	15 ⁴	70-130 ⁴
1,2,3,7,8,9-HxCDD	50	15 ⁴	70-130 ⁴
1,2,3,4,7,8-HxCDF	50	15 ⁴	70-130 ⁴
1,2,3,6,7,8-HxCDF	50	15 ⁴	70-130 ⁴
2,3,4,6,7,8-HxCDF	50	15 ⁴	70-130 ⁴
1,2,3,7,8,9-HxCDF	50	15 ⁴	70-130 ⁴
1,2,3,4,6,7,8-HpCDD	50	15 ⁴	70-130 ⁴
1,2,3,4,6,7,8-HpCDF	50	15 ⁴	70-130 ⁴
1,2,3,4,7,8,9-HpCDF	50	15 ⁴	70-130 ⁴
OCDD	100	15 ⁴	70-130 ⁴
OCDF	100	15 ⁴	70-130 ⁴

Notes:

- 1 All specifications are given as concentration in the final extract, assuming a 20-μL volume.
- 2 s = standard deviation of the percent recovery
- 3 X = average percent recovery
- 4 In-house generated historical control-limits can be used in place of the specified limit.

Table 11
Laboratory Control Sample (LCS/OPR) Spiking Solution Component Concentrations and Acceptance Limits

Analyte	LCS Solution Conc. (ng/mL) ¹	Final Extract Conc (ng/mL) ²	1613B OPR Conc (ng/mL) ²	8290, 23, 0023A, TO-9A Recovery (%Rec)
2,3,7,8-TCDD	0.2	10	6.7-15.8	70-130 ⁴
2,3,7,8-TCDF	0.2	10	7.5-15.8	70-130 ⁴
1,2,3,7,8-PeCDD	1.0	50	35-71	70-130 ⁴
1,2,3,7,8-PeCDF	1.0	50	40-67	70-130 ⁴
2,3,4,7,8-PeCDF	1.0	50	34-80	70-130 ⁴
1,2,3,4,7,8-HxCDD	1.0	50	35-82	70-130 ⁴
1,2,3,6,7,8-HxCDD	1.0	50	38-67	70-130 ⁴
1,2,3,7,8,9-HxCDD	1.0	50	32-81	70-130 ⁴
1,2,3,4,7,8-HxCDF	1.0	50	36-67	70-130 ⁴
1,2,3,6,7,8-HxCDF	1.0	50	42-65	70-130 ⁴
2,3,4,6,7,8-HxCDF	1.0	50	35-78	70-130 ⁴
1,2,3,7,8,9-HxCDF	1.0	50	39-65	70-130 ⁴
1,2,3,4,6,7,8-HpCDD	1.0	50	35-70	70-130 ⁴
1,2,3,4,6,7,8-HpCDF	1.0	50	41-61	70-130 ⁴
1,2,3,4,7,8,9-HpCDF	1.0	50	39-69	70-130 ⁴
OCDD	2.0	100	78-144	70-130 ⁴
OCDF	2.0	100	63-170	70-130 ⁴
<u>Tetras Only</u>				
2,3,7,8-TCDD	0.2	10	7.3-14.6	70-130 ⁴
2,3,7,8-TCDF	0.2	10	8.0-14.7	70-130 ⁴

Notes:

- 1 1.0 mL of this solution is added to the OPR sample before extraction (see section 7.11.1).
- 2 The final extract concentration is based on an extract volume of 20-μL.
- 3 Spike concentrations are based on a 1.0 L extraction for Water, 10.0g extraction for Solids, and entire sample extraction for Air/Wipe samples.
- 4 In-house generated historical control-limits can be used in place of the specified limit.

Table 12**Method – 8290/8290A****Matrix Spike and Matrix Spike Duplicate Sample (MS/MSD) Spiking Solution Component Concentrations and Acceptance Limits¹**

Analyte	LCS Solution Conc. (ng/mL)²	Final Extract Conc (ng/mL)³	8290 Recovery (%Rec)	8290 Precision (RPD)
2,3,7,8-TCDD	0.2	10	70-130 ⁴	±15 ⁴
2,3,7,8-TCDF	0.2	10	70-130 ⁴	±15 ⁴
1,2,3,7,8-PeCDD	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,7,8-PeCDF	1.0	50	70-130 ⁴	±15 ⁴
2,3,4,7,8-PeCDF	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,4,7,8-HxCDD	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,6,7,8-HxCDD	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,7,8,9-HxCDD	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,4,7,8-HxCDF	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,6,7,8-HxCDF	1.0	50	70-130 ⁴	±15 ⁴
2,3,4,6,7,8-HxCDF	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,7,8,9-HxCDF	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,4,6,7,8-HpCDD	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,4,6,7,8-HpCDF	1.0	50	70-130 ⁴	±15 ⁴
1,2,3,4,7,8,9-HpCDF	1.0	50	70-130 ⁴	±15 ⁴
OCDD	2.0	100	70-130 ⁴	±15 ⁴
OCDF	2.0	100	70-130 ⁴	±15 ⁴

Notes:

- 1 If insufficient sample exists for MS/MSD analysis, these limits apply to LCS/LCSD samples.
- 2 mL of this solution is added to the OPR sample before extraction (see section 7.11.2).
- 3 The final extract concentration is based on an extract volume of 20-μL.
- 4 In-house generated historical control-limits can be used in place of the specified limit.

Table 13
Methods – 1613B and 8290/8290A
Internal Standard Spiking Solution Component Concentrations and Acceptance Limits

Labeled Analyte	Solution Conc (ng/mL) ¹	Test Conc. (ng/mL) ²	1613B OPR Conc (ng/mL) ²	1613B Sample Conc (ng/mL) ²	8290 Recovery (%Rec)
¹³ C ₁₂ -2,3,7,8-TCDD	1.0	50	10.0-87.5	12.5-82.0	40-135
¹³ C ₁₂ -2,3,7,8-TCDF	1.0	50	11.0-76.0	12.0-84.5	40-135
¹³ C ₁₂ -1,2,3,7,8-PeCDD	1.0	50	10.5-113.5	12.5-90.5	40-135
¹³ C ₁₂ -1,2,3,7,8-PeCDF	1.0	50	10.5-96.0	12.0-92.5	40-135
¹³ C ₁₂ -2,3,4,7,8-PeCDF	1.0	50	6.5-164.0	10.5-89.0	40-135
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	1.0	50	10.5-96.5	16.0-70.5	40-135
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	1.0	50	12.5-81.5	14.0-65.0	40-135
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	1.0	50	9.5-101.0	13.0-76.0	40-135
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	1.0	50	10.5-79.5	13.0-61.5	40-135
¹³ C ₁₂ -2,3,4,6,7,8-HxCDF	1.0	50	11.0-88.0	14.0-68.0	40-135
¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	1.0	50	8.5-102.5	14.5-73.5	40-135
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	1.0	50	13.0-83.0	11.5-70.0	40-135
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	1.0	50	10.5-79.0	14.0-71.5	40-135
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	1.0	50	10.0-93.0	13.0-69.0	40-135
¹³ C ₁₂ -OCDD	2.0	100	13.0-198.5	17.0-157	40-135
<u>Tetras Only</u>					
¹³ C ₁₂ -2,3,7,8-TCDD	1.0	50	12.5-70.5	15.5-68.5	40-135
¹³ C ₁₂ -2,3,7,8-TCDF	1.0	50	13.0-63.0	14.5-70.0	40-135

Notes:

- 1.0 mL of the Internal Standard Spiking Solution is added to each sample, method blank and LCS/OPR sample prior to extraction (see section 7.11.3).
- Specifications given as concentration in the final extract, assuming a 20-μL volume

Table 14
Method – 1613B
Cleanup Standard Spiking Solution Component Concentrations and Acceptance Limits

Labeled Analyte	Solution Conc (ng/mL) ¹	Test Conc. (ng/mL) ²	1613B OPR Conc (ng/mL) ²	1613B Sample Conc (ng/mL) ²	1613B OPR Tetra Only Conc (ng/mL) ²	1613B Sample Tetra Only Conc (ng/mL) ²
³⁷ Cl ₄ -2,3,7,8-TCDD	0.2	10	3.1-19.1	3.5-19.7	3.7-15.8	4.2-16.4

Notes:

- 1.0 mL of the Cleanup Standard Spiking Solution is added to each sample, method blank and LCS/OPR sample prior to cleanup (see section 7.11.4).
- Specifications given as concentration in the final extract, assuming a 20-μL volume

Table 15
Methods – 23, 0023A, and TO-9A
Internal Standard Spiking Solution Component Concentrations and Acceptance Limits

Labeled Analyte	Solution Conc (ng/mL)¹	Test Conc. (ng/mL)²	23 Recovery (%Rec)	0023A Recovery (%Rec)	TO-9A Recovery (%Rec)
¹³ C ₁₂ -2,3,7,8-TCDD	1.0	50	40-130	40-135	50-120
¹³ C ₁₂ -2,3,7,8-TCDF	1.0	50	40-130	40-135	50-120
¹³ C ₁₂ -1,2,3,7,8-PeCDD	1.0	50	40-130	40-135	50-120
¹³ C ₁₂ -1,2,3,7,8-PeCDF	1.0	50	40-130	40-135	50-120
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	1.0	50	40-130	40-135	50-120
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	1.0	50	40-130	40-135	50-120
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	1.0	50	25-130	40-135	40-120
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	1.0	50	25-130	40-135	40-120
¹³ C ₁₂ -OCDD	2.0	100	25-130	40-135	40-120

Notes:

- 1.0 mL of the Internal Standard Spiking Solution is added to each sample, method blank and LCS/OPR sample prior to extraction (see section 7.11.3).
- Specifications given as concentration in the final extract, assuming a 20-μL volume

Table 16
Methods – 23, 0023A, and TO-9A
Surrogate Standard Spiking Solution Component Concentrations and Acceptance Limits

Labeled Analyte	Solution Conc (ng/mL)¹	Test Conc. (ng/mL)²	23 Recovery (%Rec)	0023A Recovery (%Rec)	TO-9A Recovery (%Rec)
³⁷ Cl ₄ -2,3,7,8-TCDD	20	100	70-130	70-130	50-120
¹³ C ₁₂ -2,3,4,7,8-PeCDF	20	100	70-130	70-130	50-120
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	20	100	70-130	70-130	50-120
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	20	100	70-130	70-130	50-120
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	20	100	70-130	70-130	40-120

Notes:

- 100 μL of the Surrogate Standard Spiking Solution is added to each sample train prior to sampling (see section 7.11.5).
- Specifications given as concentration in the final extract, assuming a 20-μL volume

Table 17
Methods – All
Recovery Standard Spiking Solution Component Concentrations

Labeled Analyte	Solution Conc (µg/mL) ¹	Test Conc. (ng/mL) ²
¹³ C ₁₂ -1,2,3,4-TCDD	0.1	100
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD	0.1	100

Notes:

- 20 µL of the Recovery Standard Spiking Solution is added to each sample, method blank and LCS/OPR sample prior to analysis (see section 7.11.8).
- Specifications given as concentration in the final extract, assuming a 20-µL volume

Table 18
Rtx-5/DB-5 Column Window Defining Standard Mixture Components

Congener	First Eluted	Last Eluted
TCDF	1,3,6,8-	1,2,8,9-
TCDD	1,3,6,8-	1,2,8,9-
PeCDF	1,3,4,6,8-	1,2,3,8,9-
PeCDD	1,2,4,6,8-/1,2,4,7,9-	1,2,3,8,9-
HxCDF	1,2,3,4,6,8-	1,2,3,4,8,9-
HxCDD	1,2,4,6,7,9-/1,2,4,6,8,9-	1,2,3,4,6,7-
HpCDF	1,2,3,4,6,7,8-	1,2,3,4,7,8,9-
HpCDD	1,2,3,4,6,7,9-	1,2,3,4,6,7,8-

Table 19
Rtx-5 (DB-5) Column Performance Standard Mixture Components

Isomer
1,2,3,7/1,2,3,8-TCDD
1,2,3,9-TCDD
2,3,7,8-TCDD

Table 20

DB-225 (Rtx-225) Column Performance Standard Mixture Components

Isomer
2,3,4,7-TCDF
2,3,7,8-TCDF
1,2,3,9-TCDF

Table 21

Ions Monitored for HRGC/HRMS Analysis of PCDDs and PCDFs

Descriptor	Accurate Mass ¹	Ion ID	Elemental Composition	Analyte
1	292.9825	LOCK	C ₇ F ₁₁	PFK
	303.9016	M	C ₁₂ H ₄ ³⁵ Cl ₄ 0	TCDF
	305.8987	M+2	C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ Cl 0	TCDF
	315.9419	M	¹³ C ₁₂ H ₄ ³⁵ Cl ₄ 0	TCDF (S)
	317.9389	M+2	¹³ C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ Cl 0	TCDF (S)
	319.8965	M	C ₁₂ H ₄ ³⁵ Cl ₄ 0 ₂	TCDD
	321.8936	M+2	C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ Cl 0 ₂	TCDD
	327.8847	M	C ₁₂ H ₄ ³⁷ Cl ₄ 0 ₂	TCDD
	331.9368	M	¹³ C ₁₂ H ₄ ³⁵ Cl ₄ 0 ₂	TCDD (S)
	333.9338	M+2	¹³ C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ Cl 0 ₂	TCDD (S)
	342.9792	QC	C ₈ F ₁₃	PFK
	375.8364	M+2	C ₁₂ H ₄ ³⁵ Cl ₅ ³⁷ Cl 0	HxCDDPE
2	330.9792	LOCK	C ₇ F ₁₃	PFK
	339.8597	M+2	C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ Cl 0	PeCDF
	341.8567	M+4	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ 0	PeCDF
	351.9000	M+2	¹³ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ Cl 0	PeCDF (S)
	353.8970	M+4	¹³ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ 0	PeCDF (S)
	355.8546	M+2	C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ Cl 0 ₂	PeCDD
	357.8516	M+4	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ 0 ₂	PeCDD
	367.8949	M+2	¹³ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ Cl 0 ₂	PeCDD (S)
	369.8919	M+4	¹³ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ 0 ₂	PeCDD (S)
	380.9760	QC	C ₈ F ₁₅	PFK
	409.7974	M+2	C ₁₂ H ₃ ³⁵ Cl ₆ ³⁷ Cl 0	HpCDPE
3	373.8208	M+2	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ Cl 0	HxCDF
	375.8178	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ 0	HxCDF
	380.9760	LOCK	C ₈ F ₁₅	PFK
	383.8639	M	¹³ C ₁₂ H ₂ ³⁵ Cl ₆ 0	HxCDF (S)
	385.8610	M+2	¹³ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ Cl 0	HxCDF (S)
	389.8156	M+2	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ Cl 0 ₂	HxCDD
	391.8127	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ 0 ₂	HxCDD
	401.8559	M+2	¹³ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ Cl 0 ₂	HxCDD (S)
	403.8529	M+4	¹³ C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ 0 ₂	HxCDD (S)
	404.9760	QC	C ₁₀ F ₁₅	PFK
	445.7555	M+4	C ₁₂ H ₂ ³⁵ Cl ₆ ³⁷ Cl ₂ 0	OCDPE

Table 21 (Continued)
Ions Monitored for HRGC/HRMS Analysis of PCDDs and PCDFs

Descriptor	Accurate Mass ¹	Ion ID	Elemental Composition	Analyte
4	404.9760	LOCK	C ₁₀ F ₁₅	PFK
	407.7818	M+2	C ₁₂ H ³⁵ Cl ₆ ³⁷ Cl 0	HpCDF
	409.7788	M+4	C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ 0	HpCDF
	417.8250	M	¹³ C ₁₂ H ³⁵ Cl ₇ 0	HpCDF (S)
	419.8220	M+2	¹³ C ₁₂ H ³⁵ Cl ₆ ³⁷ Cl 0	HpCDF (S)
	423.7767	M+2	C ₁₂ H ³⁵ Cl ₆ ³⁷ Cl 0 ₂	HpCDD
	425.7737	M+4	C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ 0 ₂	HpCDD
	435.8169	M+2	¹³ C ₁₂ H ³⁵ Cl ₆ ³⁷ Cl 0 ₂	HpCDD (S)
	437.8140	M+4	¹³ C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ 0 ₂	HpCDD (S)
	442.9728	QC	C ₁₀ F ₁₇	PFK
	479.7165	M+4	C ₁₂ H ³⁵ Cl ₇ ³⁷ Cl ₂ 0	NCDPE
5	430.9728	LOCK	C ₉ F ₁₇	PFK
	441.7428	M+2	C ₁₂ ³⁵ Cl ₇ ³⁷ Cl 0	OCDF
	443.7399	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ 0	OCDF
	457.7377	M+2	C ₁₂ ³⁵ Cl ₇ ³⁷ Cl 0 ₂	OCDD
	459.7348	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ 0 ₂	OCDD
	469.7780	M+2	¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ Cl 0 ₂	OCDD (S)
	471.7750	M+4	¹³ C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ 0 ₂	OCDD (S)
	480.9696	QC	C ₁₀ F ₁₉	PFK
	513.6775	M+4	C ₁₂ ³⁵ Cl ₈ ³⁷ Cl ₂ 0	DCDPE

Notes:

1 Nuclidic masses used:

H = 1.007825

O = 15.994915

C = 12.00000

³⁵Cl = 34.968853¹³C = 13.003355³⁷Cl = 36.965903

F = 18.9984

Table 22**Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs**

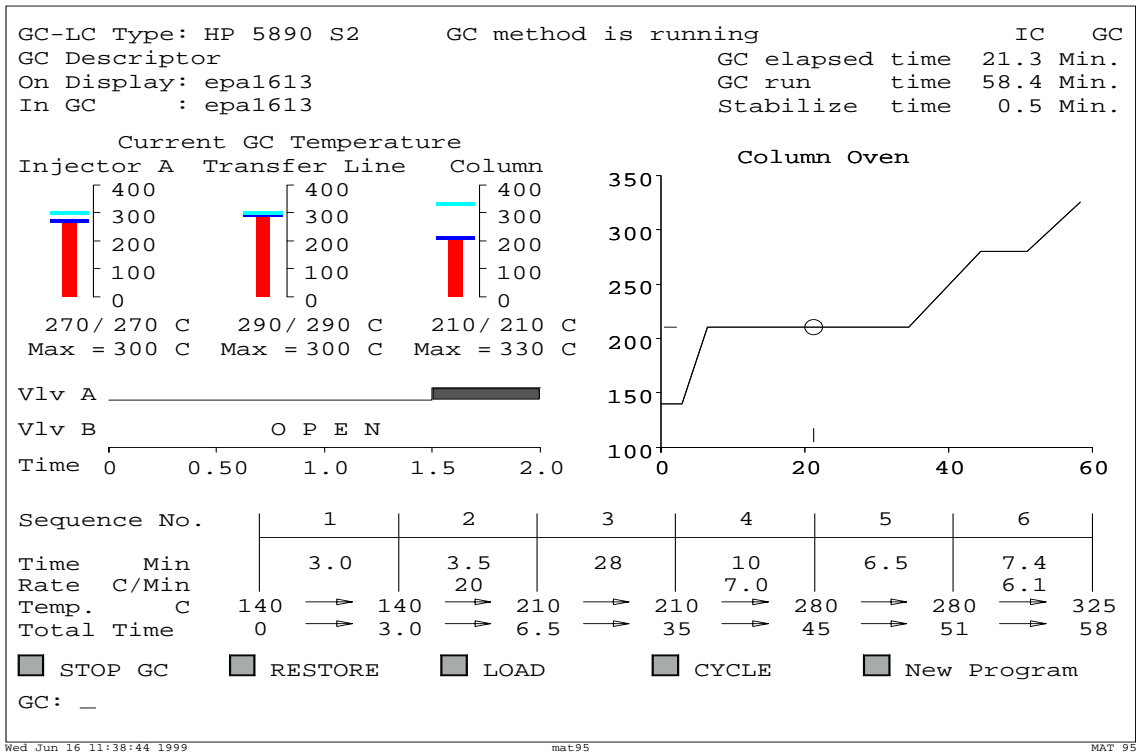
Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6 ¹	M/M+2	0.51	0.43	0.59
7	M+2/M+4	1.04/1.05 ³	0.88	1.20
7 ²	M/M+2	0.44	0.37	0.51
8	M+2/M+4	0.89	0.76	1.02

Notes:

- 1 Used for ¹³C-HxCDF (IS).
- 2 Used for ¹³C-HpCDF (IS).
- 3 Method 1613B Theoretical Ratio

Figure 1

Rtx-5 Recommended GC Operating Conditions



DB-225 Recommended GC Operating Conditions

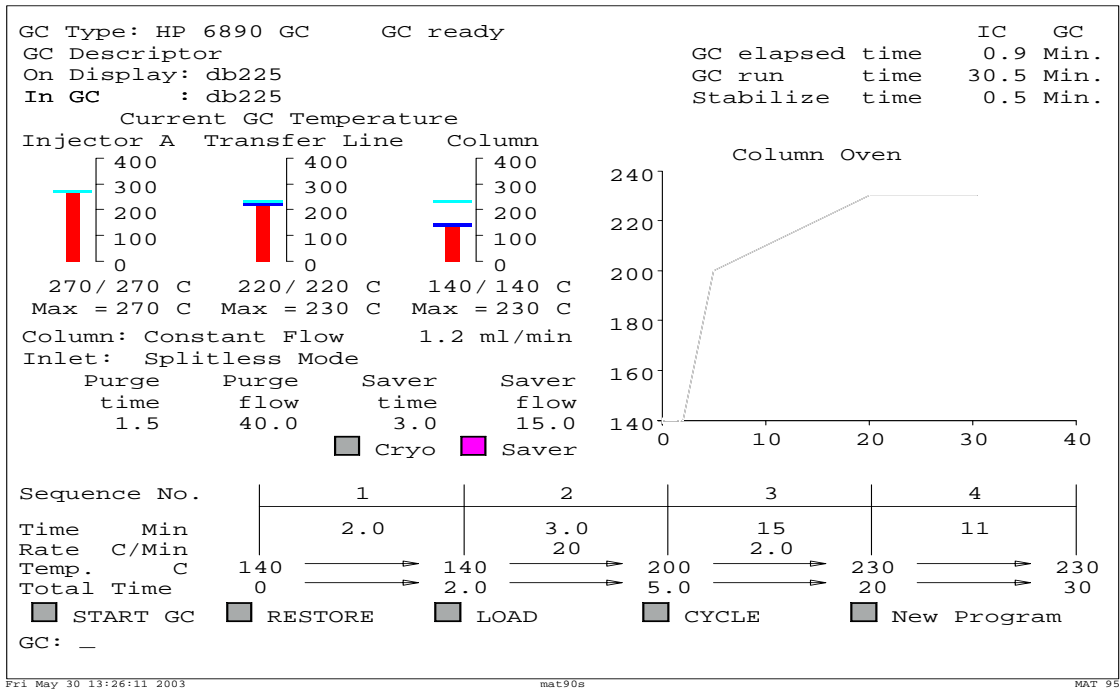


Figure 2

Rtx-5 Recommended MID Descriptors

MID Set Up Parameters					MID Masses for Time Window 1				
MID File	epal613				#	mass	F	int	gr time(ms)
Measure/lock ratio (X)	1				1	292.9825	1	10	1 8.19
Set Damping relay (T)	FALSE				2	303.9016	1	1	81.92
Width first lock (A)	0.20 amu				3	305.8987	1	1	81.92
Electric jump time (E)	10 ms				4	315.9419	1	1	81.92
Magnetic jump time (D)	60 ms				5	317.9389	1	1	81.92
Offset (O)	100 cts				6	319.8965	1	1	81.92
Electric range (R)	300 %				7	321.8936	1	1	81.92
Sweep peak width (W)	3.00				8	327.8847	1	1	81.92
Acq mode (C P)	Cent mode				9	331.9368	1	1	81.92
MID mode (J M L N)	Lock mode				10	333.9338	1	1	81.92
MID Time Windows					11	342.9792	c	10	1 8.19
#	Start	Measure	End	Cycletime	12	375.8364	1	1	81.92
1	8:00	28:12	36:12 min	1.00 sec	13				
2	36:12	7:28	43:40 min	1.00 sec	14				
3	43:40	5:49	49:30 min	1.00 sec	15				
4	49:30	5:00	54:30 min	1.00 sec	16				
5	54:30	3:50	58:20 min	1.00 sec	17				
6					18				
7					19				
8					20				
9					21				
<input type="checkbox"/> Clear Menu <input type="checkbox"/> Clear Times <input type="checkbox"/> Clear Masses <input type="checkbox"/> Stop MID <input type="checkbox"/> SAVE <input type="checkbox"/> Main					22				
MID: _					23				
					24				

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MID Set Up Parameters					MID Masses for Time Window 2				
MID File	epal613				#	mass	F	int	gr time(ms)
Measure/lock ratio (X)	1				1	330.9792	1	10	1 8.19
Set Damping relay (T)	FALSE				2	339.8597	1	1	91.48
Width first lock (A)	0.20 amu				3	341.8567	1	1	91.48
Electric jump time (E)	10 ms				4	351.9000	1	1	91.48
Magnetic jump time (D)	60 ms				5	353.8970	1	1	91.48
Offset (O)	100 cts				6	355.8546	1	1	91.48
Electric range (R)	300 %				7	357.8516	1	1	91.48
Sweep peak width (W)	3.00				8	367.8949	1	1	91.48
Acq mode (C P)	Cent mode				9	369.8919	1	1	91.48
MID mode (J M L N)	Lock mode				10	380.9760	c	10	1 8.19
MID Time Windows					11	409.7974	1	1	91.48
#	Start	Measure	End	Cycletime	12				
1	8:00	28:12	36:12 min	1.00 sec	13				
2	36:12	7:28	43:40 min	1.00 sec	14				
3	43:40	5:49	49:30 min	1.00 sec	15				
4	49:30	5:00	54:30 min	1.00 sec	16				
5	54:30	3:50	58:20 min	1.00 sec	17				
6					18				
7					19				
8					20				
9					21				
<input type="checkbox"/> Clear Menu <input type="checkbox"/> Clear Times <input type="checkbox"/> Clear Masses <input type="checkbox"/> Stop MID <input type="checkbox"/> SAVE <input type="checkbox"/> Main					22				
MID: _					23				
					24				

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Figure 2 Continued

Rtx-5 Recommended MID Descriptors

MID Set Up Parameters					MID Masses for Time Window 3					
MID File	epal613				#	mass	F	int	gr	time(ms)
Measure/lock ratio (X)	1				1	373.8208	1	1	1	91.48
Set Damping relay (T)	FALSE				2	375.8178	1	1	1	91.48
Width first lock (A)	0.20 amu				3	380.9760	1	10	1	8.19
Electric jump time (E)	10 ms				4	383.8639	1	1	1	91.48
Magnetic jump time (D)	60 ms				5	385.8610	1	1	1	91.48
Offset (O)	100 cts				6	389.8156	1	1	1	91.48
Electric range (R)	300 %				7	391.8127	1	1	1	91.48
Sweep peak width (W)	3.00				8	401.8559	1	1	1	91.48
Acq mode (C P)	Cent mode				9	403.8529	1	1	1	91.48
MID mode (J M L N)	Lock mode				10	404.9760	c	10	1	8.19
MID Time Windows					11	445.7555	1	1	1	91.48
#	Start	Measure	End	Cycletime	12					
1	8:00	28:12	36:12 min	1.00 sec	13					
2	36:12	7:28	43:40 min	1.00 sec	14					
3	43:40	5:49	49:30 min	1.00 sec	15					
4	49:30	5:00	54:30 min	1.00 sec	16					
5	54:30	3:50	58:20 min	1.00 sec	17					
6					18					
7					19					
8					20					
9					21					
<input type="checkbox"/> Clear Menu					22					
<input type="checkbox"/> Clear Times					23					
<input type="checkbox"/> Clear Masses					24					
<input type="checkbox"/> Stop MID					<input type="checkbox"/> Lock Mass <input type="checkbox"/> Cali Mass					
<input type="checkbox"/> SAVE										
<input type="checkbox"/> Main										
MID: _										

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MID Set Up Parameters					MID Masses for Time Window 4					
MID File	epal613				#	mass	F	int	gr	time(ms)
Measure/lock ratio (X)	1				1	404.9760	1	10	1	8.19
Set Damping relay (T)	FALSE				2	407.7818	1	1	1	91.48
Width first lock (A)	0.20 amu				3	409.7788	1	1	1	91.48
Electric jump time (E)	10 ms				4	417.8250	1	1	1	91.48
Magnetic jump time (D)	60 ms				5	419.8220	1	1	1	91.48
Offset (O)	100 cts				6	423.7767	1	1	1	91.48
Electric range (R)	300 %				7	425.7737	1	1	1	91.48
Sweep peak width (W)	3.00				8	435.8169	1	1	1	91.48
Acq mode (C P)	Cent mode				9	437.8140	1	1	1	91.48
MID mode (J M L N)	Lock mode				10	442.9728	c	10	1	8.19
MID Time Windows					11	479.7165	1	1	1	91.48
#	Start	Measure	End	Cycletime	12					
1	8:00	28:12	36:12 min	1.00 sec	13					
2	36:12	7:28	43:40 min	1.00 sec	14					
3	43:40	5:49	49:30 min	1.00 sec	15					
4	49:30	5:00	54:30 min	1.00 sec	16					
5	54:30	3:50	58:20 min	1.00 sec	17					
6					18					
7					19					
8					20					
9					21					
<input type="checkbox"/> Clear Menu					22					
<input type="checkbox"/> Clear Times					23					
<input type="checkbox"/> Clear Masses					24					
<input type="checkbox"/> Stop MID					<input type="checkbox"/> Lock Mass <input type="checkbox"/> Cali Mass					
<input type="checkbox"/> SAVE										
<input type="checkbox"/> Main										
MID: _										

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Figure 2 Continued

Rtx-5 Recommended MID Descriptors

MID Set Up Parameters					MID Masses for Time Window 5				
MID File	epal613				#	mass	F	int	gr time(ms)
Measure/lock ratio (X)	1				1	430.9728	1	10	1 10.92
Set Damping relay (T)	FALSE				2	441.7428	1	1	120.15
Width first lock (A)	0.20 amu				3	443.7399	1	1	120.15
Electric jump time (E)	10 ms				4	457.7377	1	1	120.15
Magnetic jump time (D)	60 ms				5	459.7348	1	1	120.15
Offset (O)	100 cts				6	469.7780	1	1	120.15
Electric range (R)	300 %				7	471.7750	1	1	120.15
Sweep peak width (W)	3.00				8	480.9696	c	10	1 10.92
Acq mode (C P)	Cent mode				9	513.6775	1	1	120.15
MID mode (J M L N)	Lock mode				10				
MID Time Windows					11				
#	Start	Measure	End	Cycletime	12				
1	8:00	28:12	36:12 min	1.00 sec	13				
2	36:12	7:28	43:40 min	1.00 sec	14				
3	43:40	5:49	49:30 min	1.00 sec	15				
4	49:30	5:00	54:30 min	1.00 sec	16				
5	54:30	3:50	58:20 min	1.00 sec	17				
6					18				
7					19				
8					20				
9					21				
<input type="checkbox"/> Clear Menu <input type="checkbox"/> Clear Times <input type="checkbox"/> Clear Masses					22				
<input type="checkbox"/> Stop MID <input type="checkbox"/> SAVE <input type="checkbox"/> Main					23				
MID: _					24				
						<input type="checkbox"/>	Lock Mass	<input type="checkbox"/>	Cali Mass

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DB-225 Recommended MID Descriptor

MID Set Up Parameters					MID Masses for Time Window 1				
MID File	db225				#	mass	F	int	gr time(ms)
Measure/lock ratio (X)	1				1	292.9825	1	10	1 8.19
Set Damping relay (T)	TRUE				2	303.9016	1	1	81.92
Width first lock (A)	0.20 amu				3	305.8987	1	1	81.92
Electric jump time (E)	10 ms				4	315.9419	1	1	81.92
Magnetic jump time (D)	60 ms				5	317.9389	1	1	81.92
Offset (O)	100 cts				6	319.8965	1	1	81.92
Electric range (R)	300 %				7	321.8936	1	1	81.92
Sweep peak width (W)	3.00				8	327.8847	1	1	81.92
Acq mode (C P)	Cent mode				9	331.9368	1	1	81.92
MID mode (J M L N)	Lock mode				10	333.9338	1	1	81.92
MID Time Windows					11	342.9792	c	10	1 8.19
#	Start	Measure	End	Cycletime	12	375.8364	1	1	81.92
1	8:00	22:30	30:30 min	1.00 sec	13				
2					14				
3					15				
4					16				
5					17				
6					18				
7					19				
8					20				
9					21				
<input type="checkbox"/> Clear Menu <input type="checkbox"/> Clear Times <input type="checkbox"/> Clear Masses					22				
<input type="checkbox"/> Start MID <input type="checkbox"/> RESTORE <input type="checkbox"/> Main					23				
MID: _					24				
						<input type="checkbox"/>	Lock Mass	<input type="checkbox"/>	Cali Mass

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Example Sample Prep Benchsheets

Test America Knoxville - Specialty Organics Group - Sample Tracking Sheet - Organic Preparation Group

QC Batch No: 9055311 Internal Std. Spiked By: _____ Verified By: _____ Date: _____
 Sodium Sulfate Lot: _____ Native LCS/D Spiked By: _____ Verified By: _____ Date: _____
 (only for Waters) Native MS/D Spiked By: _____ Verified By: _____ Date: _____

Work Order Number	Filled Y/N?	Vol of Sampling Surrogate (uL)	Soxhlet Started (Date/Time)	Soxhlet Finished (Date/Time)	Blow Down (Initial/Date)	Recovery Standard (Book/Page)	Recovery Std Vol (uL)	Vol Delivered (uL)	Comments
K7FJX1AC									
K7FJ41AC									
K7FJ51AC									
K7FJ61AC									
K7FJ81AC									
K7FJ91AC									
K7FKA1AC									
K7MH41AA	B								
K7MH41AC	C								
K7MH41AD	L								

Balance ID: _____ Volume of Alt Standard: _____
 Split Ratios: Post I.S. _____ Post F.C. _____ Post C.S. _____ Post R.S. _____

Blank Matrix: _____ W.B. Temp _____ Lot #: _____

Extraction Solvent: _____

Comments: _____

Figure 3 Continued
Example Sample Prep Benchsheets

QC Batch No: 9055311

Lot #s

Test America Knoxville

Specialty Organics Group - Cleanup Tracking Sheet - Organic Preparation Group

Cleanup Std. Spiked By: _____
Deact. Silica: _____
Acid Silica: _____
Alumina: _____
Sodium Sulfate: _____

Verified By: _____
MeCl2: (A) _____
Hexane: (1) _____

Date: _____
Toluene: _____
Cyclohexane: _____
Benzene: _____
Methanol: _____

Ethyl Ether _____

Work Order Number	Cleanup Spike (Book/Page)	Cleanup Spike Volume (uL)	SilicaGel (A) Alumina Col(1) (Initial/Date)	Acid/Base Wash (Initial/Date)	Carbon Column (Initial/Date) (B)	Florisil Column (Initial/Date) (2)	Mercury Cleanup (Initial/Date)	TBA Cleanup (Initial/Date)	Other Cleanup (Initial/Date) (C)	Comments
K7FJX1AC										
K7FJ41AC										
K7FJ51AC										
K7FJ61AC										
K7FJ81AC										
K7FJ91AC										
K7FKA1AC										
K7MH41AA	B									
K7MH41AC	C									
K7MH41AD	L									

Figure 3 Continued
Example Sample Prep Benchsheets

QC Batch No: **9068226** Relinquished to GC/MS By: _____ Date: _____
Received in GC/MS By: _____ Date: _____

Work Order #	Column: RTX-5				Column: RTX-5				Column: DB-225			
	Date	Instrument	Analyst	Code	Date	Instrument	Analyst	Code	Date	Instrument	Analyst	Code
K71P3 1AA												
K71P6 1AA												
K71P6 1AC				S								
K71P6 1AD				D								
K71P8 1AA												
K71QA 1AA												
K71QD 1AA												
K71QF 1AA												
K71QG 1AA												
K73FA 1AA												
K78FC 1AA				B								
K78FC 1AC				C								

Comments: _____

Figure 4

Example Sample Prep Data Review Checklist

TestAmerica Knoxville Specialty Organics Preparation Batch Review/Checklist Batch# _____

☐ KNOX-ID-0004, rev 8 ☐ KNOX-ID-0012M, rev 2 ☐ KNOX-ID-0013, rev 7 ☐ KNOX-ID-0016, rev 6

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd Level
1. Does the batch contain no more than 20 field samples? (Excluding MB, LCS, LCSD, MS, & MSD)					
2. Were the samples extracted by the proper method?					
3. Were the samples extracted within the required holding times?					
4. For waters by 1613B, if visible solids were present, were solids determined to be $\leq 1\%$?					
5. Were all project specific requirements met as noted on the Lot Checklists and Sample Worksheets?					
6. Were all required QC samples prepared & extracted with the batch at method required frequency?					
7. Were MS Run# properly assigned and samples entered on QC tracking Sheet?					
8. Were samples requested properly and request form completed, signed, and dated?					
9. Were the correct weights and volumes entered in Quantims for all samples?					
10. Were the internal standards properly spiked and the spikes verified? Were the spike solution ID and spike volumes entered correctly and verified?					
11. Were alternate standards properly spiked and the spikes verified? Were the spike solution ID and spike volumes entered correctly and verified?					
12. Were all cleanup steps properly documented by initials and date?					
13. Was the final volume checked and verified against the supplemental benchsheet and Quantims?					
14. Are the final extracts free of water, precipitates, multiple phases, and color?					
15. Were all appropriate notes and observations recorded on the prep benchsheet and in Quantims?					
16. Were all Quantims batch information completed including: <ul style="list-style-type: none"> • Batch reviewed • Correct volumes entered • Correct completion date entered • Samples released 					
17. Does the prep batch paperwork package contain all required documentation which has been properly and completely filled out, including: <ul style="list-style-type: none"> • Prep Benchsheet • Supplemental Benchsheet • Standard concentration forms or copies of logbook pages, for all IS, RS, SS, CS, Native and Alternate standards. • Lot Checklists for all lots in the batch • Sample worksheets for all samples in the batch in proper order as recorded on tracking sheet 					
18. Are all nonconformances documented appropriately and copy included with deliverable?					

ID025R11.doc 031109

Figure 4 Continued

Example Sample Data Review Checklist

TestAmerica Knoxville Dioxin GC/MS Initial Calibration Data Review / Narrative Checklist Method: 8290/8290A - KNOX-ID-0004-R8

PFK Date/Time:		Inst:		Win Filename:		Col Perf Filename:	
----------------	--	-------	--	---------------	--	--------------------	--

CS1 Filename	CS2 Filename	CS3 Filename	CS4 Filename	CS5 Filename

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd Level
1. Was the mass resolution documented before beginning the initial calibration?					
2. Was the instrument resolution >10,000 (<100 ppm) on PFK m/z 304.9824 and m/z 380.9760 (at reduced voltage)?					
3. Was the measured exact mass of m/z 380.9760 (PFK) within 5 ppm at reduced accelerating voltage?					
4. Was the Window Defining Mixture analyzed and the MID switchpoints set to encompass the retention time windows of each congener group?					
5. Was the Column Performance solution analyzed and the %Valley ≤25 for separation between 2378-TCDD/F and the closest eluting non-2378 isomer?					
6. Were the five calibration standard solutions, at the concentrations specified in Table 5 of the SOP, analyzed?					
7. Was date/time of analysis verified between analysis header and logbook as correct?					
8. Were the response factors calculated for each labeled standard and unlabeled native analyte using the SOP specified reference compound (Table 5), quantitation ions (Table 22), and formula (Section 10.2.6)?					
9. Are the relative retention times of all PCDDs/PCDFs and all labeled compounds within the limits specified in Table 3?					
10. Are %RSD ≤20% for all unlabeled native analytes?					
11. 8290, are %RSD ≤30% for all labeled internal standards?					
12. 8090A, are %RSD ≤20% for all labeled internal standards?					
13. Are all S/N ratios ≥10 for the GC signals in each EICP (extracted ion chromatographic profile) including internal standards?					
14. Are the ion abundance ratios for all labeled and unlabeled analytes within the control limits specified in Table 22 of the SOP?					
15. Was the second source (Wellington) ICV standard analyzed, and the %D calculated and within the acceptance criteria of < ±3%?					
16. If manual integrations were performed, are they clearly identified, initialed and dated?					
17. Were before/after chromatograms reviewed to determine whether the software and manual integrations were appropriate?					
18. Were manual integrations performed properly?					
19. If criteria were not met, was a NCM generated, approved by supervisor, and copy included in folder?					
20. Does the ICAL folder contain complete data in the following order? Data review checklist, a complete runlog, Avg. %RSD summary, Ratio summary, Calculation summary, PFK resolution/peak match documentation; Total RIC, EICP's and manual integration - for window and all standards, in order from low to high standard, ICV Summary Table, Calculation Summary, and chromatograms?					

Analyst:	Date:	2nd Level Reviewer :	Date:
Comments:		Comments:	

Figure 4 Continued

Example Data Review Checklist

TestAmerica Knoxville Dioxin GC/MS Continuing Calibration Review / Narrative Checklist

Method: 8290/8290A - KNOX-ID-0004-R8

Start PFK:		Ver Filename:		Inst:	
End PFK:		Win Filename:		ICAL Date:	
		Col Perf Filename:			
		End Ver Filename:			

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd Level
1. Was the mass resolution documented at both the beginning and end of the 12 hour shift?					
2. Was the instrument resolution >10,000 (<100 ppm) on PFK m/z 304.9824 and m/z 380.9760 (at reduced voltage)?					
3. Was the measured exact mass of m/z 380.9760 (PFK) within 5 ppm at reduced accelerating voltage?					
4. Was date/time of analysis verified between analysis header and logbook as correct?					
5. Was the Window Defining Mixture analyzed and the MID switchpoints set to encompass the retention time windows of each congener group?					
6. Was the Column Performance solution analyzed and the %Valley ≤25 for separation between 2378-TCDD/F and the closest eluting non-2378 isomer?					
7. Were continuing calibrations performed at the beginning and end of the 12-hour period, with successful mass resolution and GC resolution performance checks?					
8. Were the response factors calculated for each labeled standard and unlabeled native analyte using the SOP specified reference compound (Table 3), quantitation ions (Table 22), and formula (Section 10.2.6)?					
9. Are the measured RRFs for each compound within the specified control limits in Table 7 for all PCDDs/PCDFs?					
10. Are the relative retention times of all PCDDs/PCDFs and all labeled compounds within the limits specified in Table 3?					
11. Are all S/N ratios ≥10 for the GC signals in each EICP (extracted ion chromatographic profile) including internal standards?					
12. Are the ion abundance ratios for all labeled and unlabeled analytes within the control limits specified in Table 22 of the SOP?					
13. If manual integrations were performed, are they clearly identified, initialed and dated?					
14. Were before/after chromatograms reviewed to determine whether the software and manual integrations were appropriate?					
15. Were manual integrations performed properly?					
16. If criteria were not met, was a NCM generated, approved by supervisor, and copy included in folder?					
17. Does the CCAL folder contain complete data in the following order: Data review checklist, a complete runlog, CCAL summary, Ratio summary, Calculation summary, PFK resolution/peak match documentation; Total RIC, EICP's and manual integration - for window and all standards?					

Analyst:	Date:	2nd Level Reviewer :	Date:
Comments:	Comments:		

Figure 4 Continued

Example Data Review Checklist

TestAmerica Knoxville Dioxin GC/MS Data Review / Narrative Checklist LOT # _____
 Method: 8290/8290A- KNOX-ID-0004-R8 Page 1 of 1 Batch# _____

Review Items	N/A	Yes	No	Why is data reportable?	2nd Level																	
A. Initial Calibration																						
1. Was the correct ICAL used for quantitation? (Check 1-2 compounds for batch by manually calculating concentration using the ICAL avg. RF.)																						
B. Continuing Calibration																						
1. Has a Continuing Calibration Checklist been completed for each analytical batch?																						
C. Client Sample AND QC Sample Results																						
1. Were all special project requirements met?																						
2. Were the header information, prep factors, and dilution factors verified?																						
3. Is logbook date/time of analysis correct?																						
4. Sample analyses done within preparation and analytical holding time (HT)? <i>If no, list samples:</i> _____				<input type="checkbox"/> HT expired upon receipt. <input type="checkbox"/> * Client requested analysis after HT expired. <input type="checkbox"/> Re-extraction done after HT expired.																		
5. Are internal standards within QC limits specified in Table 13? <i>If no, list samples and reason (e.g., sur1):</i> <table border="0" style="width: 100%;"> <tr> <td style="width: 25%;">Sample</td> <td style="width: 25%;">Reason</td> <td style="width: 25%;">Sample</td> <td style="width: 25%;">Reason</td> </tr> <tr><td>_____</td><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td><td>_____</td></tr> </table>	Sample	Reason	Sample	Reason	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	<input type="checkbox"/> * [sup] Ion suppression due to matrix. <input type="checkbox"/> * [low] Low recovery. S/N > 10 and EDL < ML. <input type="checkbox"/> [sam] Not enough sample to re-extract. <input type="checkbox"/> [dil] Dilution showed acceptable %R. <input type="checkbox"/> [mbx] Obvious matrix interference. Further cleanup not possible. <input type="checkbox"/> * [unk] At client's request, data was flagged as estimated and released without further investigation.	
Sample	Reason	Sample	Reason																			
_____	_____	_____	_____																			
_____	_____	_____	_____																			
_____	_____	_____	_____																			
_____	_____	_____	_____																			
6. Were reported PCDD/Fs which did not meet the criteria below, properly calculated and reported as EMPCs?: <ul style="list-style-type: none"> • RT of 2378 isomers within -1 to +3 seconds of associated labeled isomer. • RT of non-2378 isomers within established first/last windows. • Both native ions maximized within ± 2 seconds. • Ion abundance ratios within the control limits specified in Table 22. • No corresponding peak at PCDFE mass. 																						
7. Were all 2378-TCDF hits \geq ML confirmed by analysis on DB-225?																						
8. Are positive results > ML within calibration range? <i>If no, list samples:</i> _____				<input type="checkbox"/> OCDD/F or non-2378 exceeded calibration range <input type="checkbox"/> Sample extracted at lowest possible volume																		
9. Are all manual integrations performed properly and clearly identified and approved?																						
10. Were before/after chromatograms reviewed to determine whether the software and manual integrations were appropriate?																						
11. Final report acceptable? (Results correct, DLs calculated correctly, units correct, IS %R correct, appropriate flags used, dilution factor correct, and extraction/analysis dates correct.)																						
12. Was a narrative prepared and all deviations noted?																						
D. Preparation/Matrix QC																						
1. LCS(OPR) done per prep batch and all analytes within the limits specified in QuantIMS reference data?				<input type="checkbox"/> * Reanalysis not possible-insufficient sample. <input type="checkbox"/> LCS %R high and affected analyte(s) were <ML in associated samples.																		
2. Method blank done per prep batch, method/instrument blank analyzed with each sequence and analytes present in the method blank \leq ML? <i>If no, list blank ID:</i> _____				<input type="checkbox"/> Sample results are > 20x higher than blank. <input type="checkbox"/> * There is no analyte > RL in the samples associated with method blank. <input type="checkbox"/> * Reanalysis not possible-insufficient sample																		
3. MS/MSD recoveries and RPDs within laboratory generated QC limits? <i>If no, list MS/MSD</i> _____				<input type="checkbox"/> LCS acceptable, indicating sample matrix effects. <input type="checkbox"/> LCS acceptable, high analyte concentration. <input type="checkbox"/> LCS acceptable, lack of sample homogeneity.																		
E. Other																						
1. Are all nonconformances documented appropriately and copy included with deliverable?																						

Analyst:	Date:	Analyst:	Date:
Comments:		Comments:	

* Such action must be taken in consultation with client.

ID022R11.doc, 031109

Figure 5
Aqueous Sample Extraction Flowchart

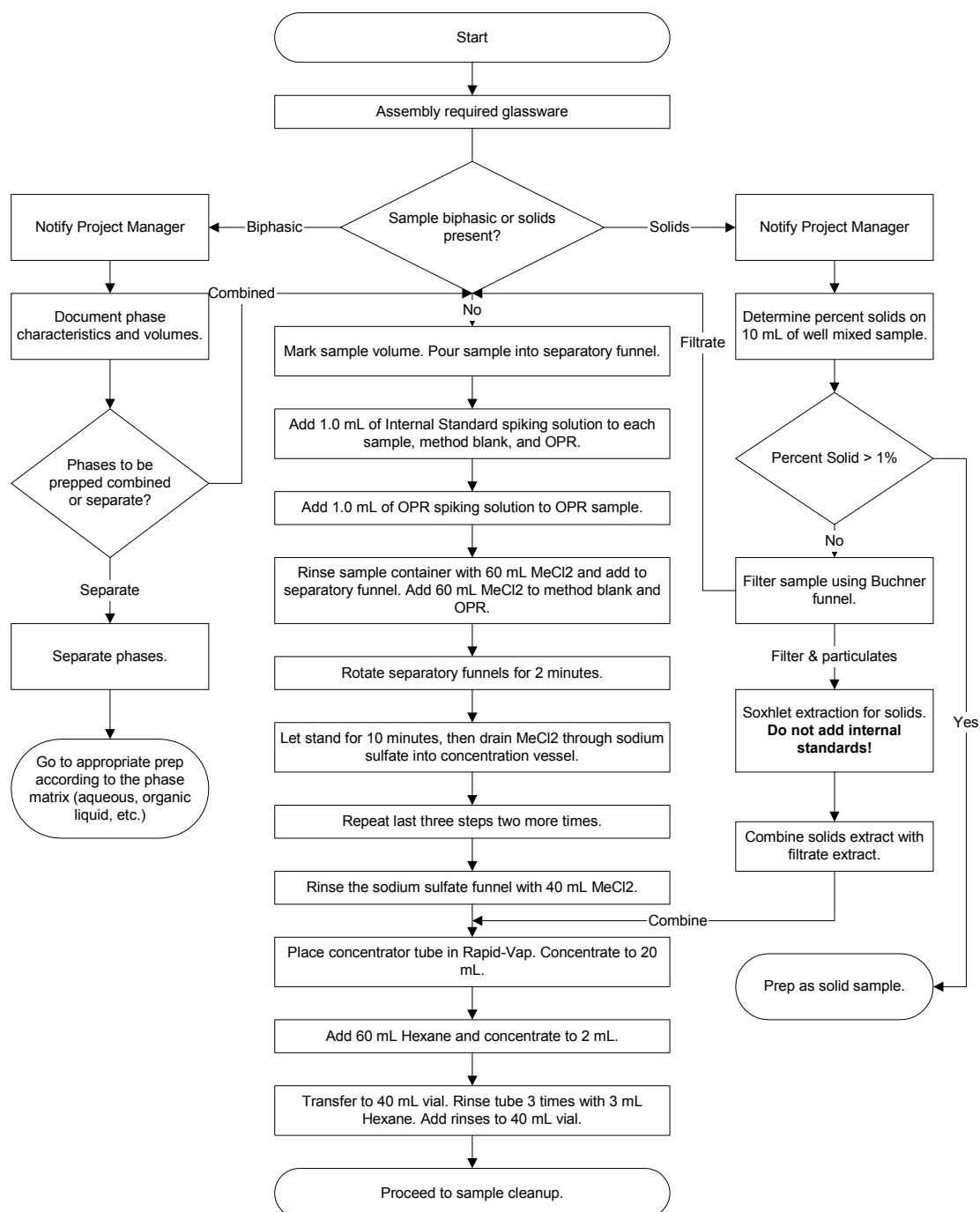


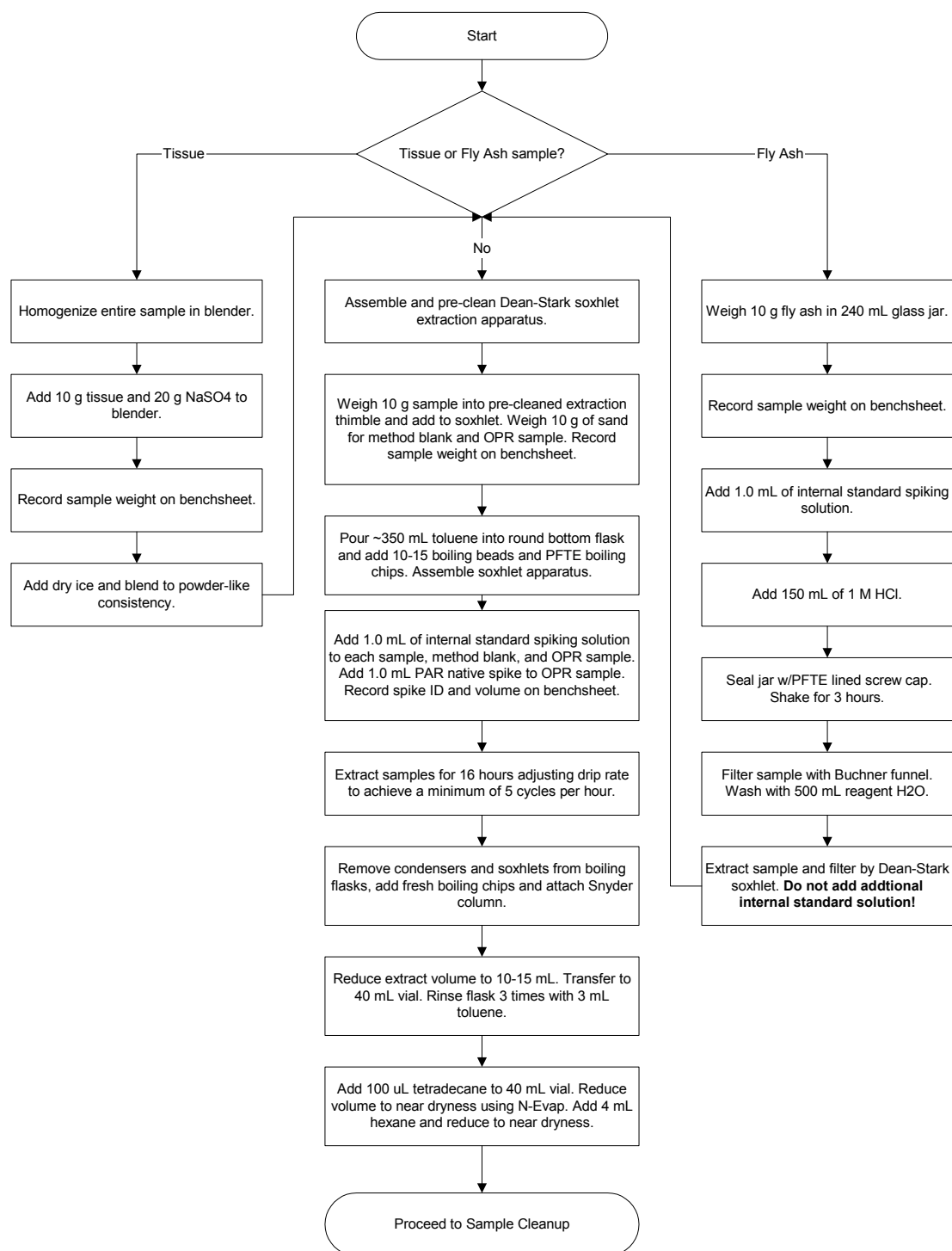
Figure 6**Solid Sample Extraction Flowchart**

Figure 7
Sample Cleanup Flowchart

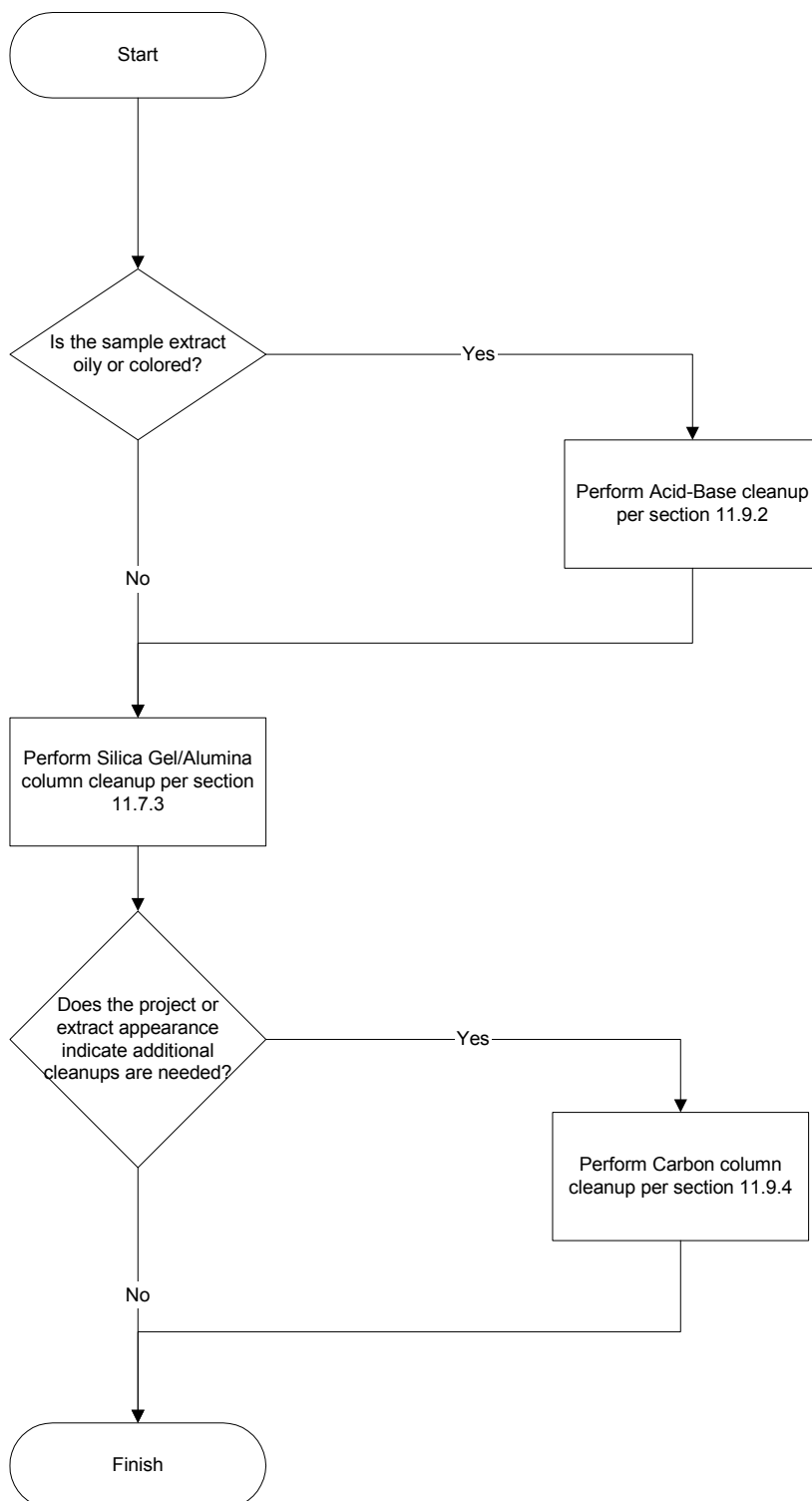
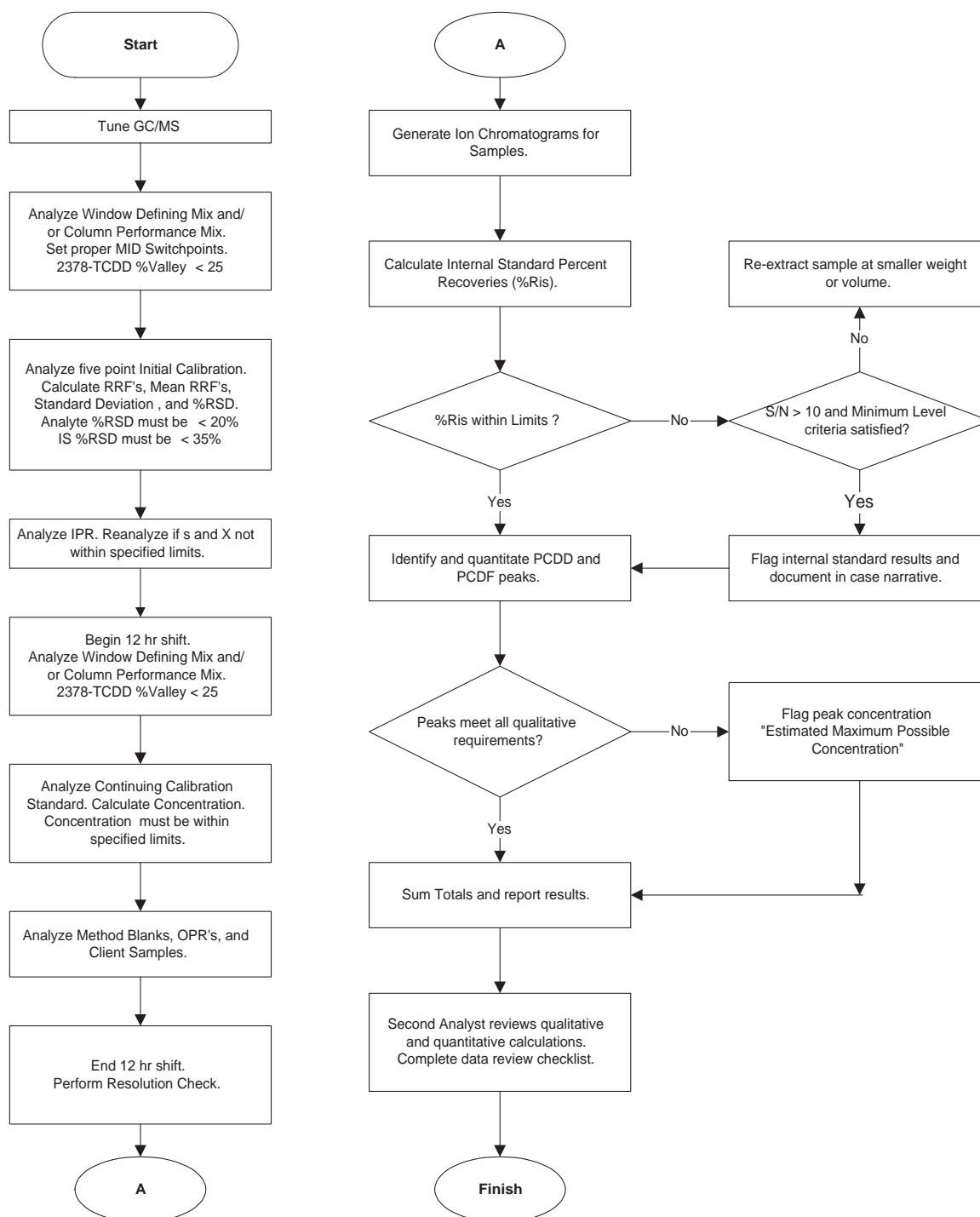


Figure 8**Analysis of PCDD's and PCDF's by HRGC/HRMS**

Appendix I Lipids Determination

1.0 This lipids determination procedure defaults to the extraction procedure for tissue as defined in the reference standard operating procedure KNOX-ID-0004, current revision, Section 11 with the following modifications:

1.1 Weigh out 20 grams of homogenized tissue and record the sample amount on the sample prep form or in a logbook. Place the 20g of homogenized tissue along with 30 grams of sodium sulfate to a laboratory blender. Blend the tissue/sodium sulfate mixture while adding small chips of dry ice to achieve a powder like consistency. Additional amounts of sodium sulfate or several more chips of dry ice may be necessary to complete the transformation of the tissue to a powder like consistency. Use a balance with a sensitivity of at least 0.01 grams and with a precision of ± 0.02 grams using a 0.1 gram NIST traceable weight.

1.2 Carefully add the mixture to a glass fiber thimble.

1.3 Assemble the Soxhlet system and secure to the lab supports.

1.4 For tissue sample, sodium sulfate and dry ice are used for method blank, LCS/OPR and LCSD. Transfer 20 ± 0.5 grams of the sodium sulfate and several small chips of dry ice into an extraction thimble. Record the blank matrix type on the bench sheet.

1.5 Pour approximately 350 mL toluene into a 500 mL round bottom flask. Place the flask in the heating mantle. Add 10-15 boiling beads and several PTFE boiling chips.

1.6 Place the extraction thimble in the glass Soxhlet extractor.

Note: Place the method blank and QC samples in random positions within the available prep positions in the hood (i.e do not use the same positions each time the method blank and QC samples are prepared).

1.7 Spike each sample with 2.0 mL of the internal standard spiking solution (see section 7.11.3) and add a small amount of glass wool if needed to the top of the extraction thimble. Record the standard solution ID and volume spiked on the sample prep sheet. Initial and date the entry.

1.7.1 Spike the LCS/OPR, LCSD (if required), and MS/MSD (8290 only) samples with 2.0 mL of the PAR native spiking solutions (see section 7.11.2) prior to adding the glass wool. Record the standard solution ID and volume spiked on the sample prep sheet. Initial and date the entry.

Note: If the volume of standard in the stock container is less than 10 mL after use, discard the remaining portion and prepare a new batch as specified in section 7.11.2).

1.8 Adjust the temperature of the heating mantle to bring the toluene in the round bottom flask to a rolling boil. There must be a steady drip from the condensers so that the solvent

completely cycles at least 5 times an hour. Record the date and time that the Soxhlet extraction was started on the bench sheet, initial and date.

1.9 Soxhlet extract the sample in the above manner for a minimum of 16 hours. At the end of the extraction period, turn off the heating mantles. Record the date and time that the Soxhlet extraction was completed on the bench sheet, initial and date.

1.10 Remove the condensers and empty the Soxhlet extractor chamber, then remove the Soxhlet extractor from the 500 mL round bottom flask.

1.11 Add several (2-3) fresh boiling chips to the flasks. Insert a three-ball macro Snyder column into the top of the 500 mL round bottom flask.

1.12 Place the 500 mL flask back into the heating mantle and reduce the extract volume to approximately 20 mL.

1.13 Transfer extract to a 40 mL vial marked at 20 mL.

1.14 Take 10 mL of the extract for lipids determination. Process the other 10 mL as necessary for method 8290.

1.15 Determination of the lipids content of sample

1.15.1 Adjust the volume of rinsings to approximately 20 mL by either adding more toluene or concentrating down the rinsing.

1.15.2 Weigh the aluminum foil weighing boat and record the weight (tare weight) on the lipid determination worksheet. Use an analytical balance capable of weighing to 0.001 grams with a precision of ± 0.002 grams using a 0.1 gram NIST traceable weight.

1.15.3 Remove 10 mL of the extract and place in the aluminum foil weighing boat. Immediately weigh the boat and extract to reduce solvent evaporation. Record the weight on the lipid determination worksheet.

1.15.4 Place the boat in the hood and allow the solvent to evaporate. Dry the sample overnight (at least 12 hours). Weigh the boat after all the solvent has evaporated. Allow the boat to air dry for one more hour and reweigh the boat. Repeat this process several times to make sure the solvent is completely removed or until constant weight is achieved (≤ 0.020 grams difference between weighings). Record the weights on the lipids determination worksheets. Enter the data into the Gravimetric Lipids Sample Analysis worksheet found in the Knx Orgprep general templates. Upload the data from the worksheet into QuantIMS.

1.15.5 The percent lipids is calculated as follows:

$$\% \text{ Lipids} = (\text{Residue Weight} / \text{Tissue Weight}) * 100$$

Where:

$$\text{Residue Weight (g)} = \text{Dry Weight} - \text{Tare Weight}$$

$$\text{Tissue Weight (g)} = \frac{(\text{Weight of Sample Extracted} * \text{Volume used for Lipid (mL)})}{\text{Total Extract Volume (mL)}}$$

“Dry Weight” in this case refers to the weight of the aluminum foil boat, containing residue, after solvent evaporation.

1.15.6 Record the 50% split of the extract on the extraction bench sheet.

1.16 Process the other 10 mL of remaining extract as necessary for method 8290, see KNOX-ID-0004, section 11 for details.

Table 1 Summary of QC Measures

Performance Area	Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank/ Instrument Blank	Concentration of target analytes, using all reagents & glassware used for sample analysis.	Minimum of 1 for each 20 samples.	\leq Reporting Limit (minimum level)	If less than 5% of sample concentrations, narrate. If greater than 5% of sample concentrations, re-extract entire batch.
Laboratory Duplicate	Precision Comparison of % Lipids	Analyze duplicate at a frequency of 1 per 20 client samples. (MS/MSD can be used)	RPD \leq 20% for %Lipids >5x RL. RPD \leq +/- 2xRL for %Lipids \leq 5x RL	Review calculations. Narrate.

Attachment I: LRMS Dioxin Screen Strategy

Analyte	10g		5X UCL 10 g 20 uL	5X UCL @ 1 g	Level 4 Samp Conc pg/g	Level 4 Screen	Level 4 Screen	Level 4 Screen	Level 4 Screen
	LCL ppt	UCL pg/g				Using 1 g 20uL pg/g	Using 1 g 500 uL pg/g	Using 1.25 g 500 uL pg/g	Using 2.5 1000 uL pg/g
2,3,7,8-TCDD	1	400	2000	20000	100	1000	25000	20000	20000
2,3,7,8-TCDF	1	400	2000	20000	100	1000	25000	20000	20000
1,2,3,7,8-PeCDD	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,7,8-PeCDF	5	2000	10000	100000	500	5000	125000	100000	100000
2,3,4,7,8-PeCDF	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,4,7,8-HxCDD	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,6,7,8-HxCDD	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,7,8,9-HxCDD	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,4,7,8-HxCDF	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,6,7,8-HxCDF	5	2000	10000	100000	500	5000	125000	100000	100000
2,3,4,6,7,8-HxCDF	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,7,8,9-HxCDF	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,4,6,7,8-HpCDD	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,4,6,7,8-HpCDF	5	2000	10000	100000	500	5000	125000	100000	100000
1,2,3,4,7,8,9-HpCDF	5	2000	10000	100000	500	5000	125000	100000	100000
OCDD	10	4000	20000	200000	1000	10000	250000	200000	200000
OCDF	10	4000	20000	200000	1000	10000	250000	200000	200000

If levels in screen are greater than the level 4 standard, do not prep. Send for 8280 analysis.

If peaks are observed at 1/5 to 1 time(s) the areas of the level 4 standard, prep at 1 g, 1/10 bench dilution.***

If peaks are observed at 1/50 to 1/5 time(s) the areas of the level 4 standard, prep at 1 g.***

If no peaks are observed, prep 10 grams.

*** In these cases, the glassware used is treated as contaminated. Post- Clean with solvent before washing.

Attachment II: Example Of Determination of Sediment Sample Extraction Amount

TestAmerica Knoxville
Determination of Sediment Sample Extraction Amounts

Prep Batch #: _____
OR Lot ID: _____ Meth. Code: _____

	Lot #	Sample Number	Work Order ID	Percent Moisture	Percent Solids	Extraction Amount (g)	Dry Weight Amount (g)	LIMS DF (GC Use Only!)
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

TestAmerica Knoxville
Gravimetric Lipids Data Worksheet

Batch #: _____

Prep Date: _____ Analyst Initials: _____ Anal Date (mm/dd/yy): _____ Reviewed by: _____

Comp Date: _____ Analyst LIMS Code: _____ Instr. ID: BIAL Date: _____

[illegible]

Spike Information:

Spike	Standard ID	Spike Vol. (mL)	Std. Conc. (mg/mL)	Work Order	Final Sample Weight (g)	True Spike Conc (% Lipids)
ILCS						

Reagents
MeCl2 Lot #
Acetone Lot #

Comments:

$$\begin{aligned} \text{Final Sample Weight (g)} &= \text{Total Sample Weight (g)} \times \text{Volume Extract Used (mL)} / \text{Total Volume Extract (mL)} \\ \text{Residue Weight (g)} &= \text{Dry Weight (g)} - \text{Tare Weight (g)} \\ \% \text{ Lipids} &= 100 \times \text{Residue Weight (g)} / \text{Final Sample Weight (g)} \\ \text{True Spike Concentration (\% Lipids)} &= 100 \times \text{Standard Concentration (mg/mL)} \times \text{Spike Volume (mL)} / \text{Final Sample Weight (g)} \times 1 \text{ g} / 1000 \text{ mg} \end{aligned}$$